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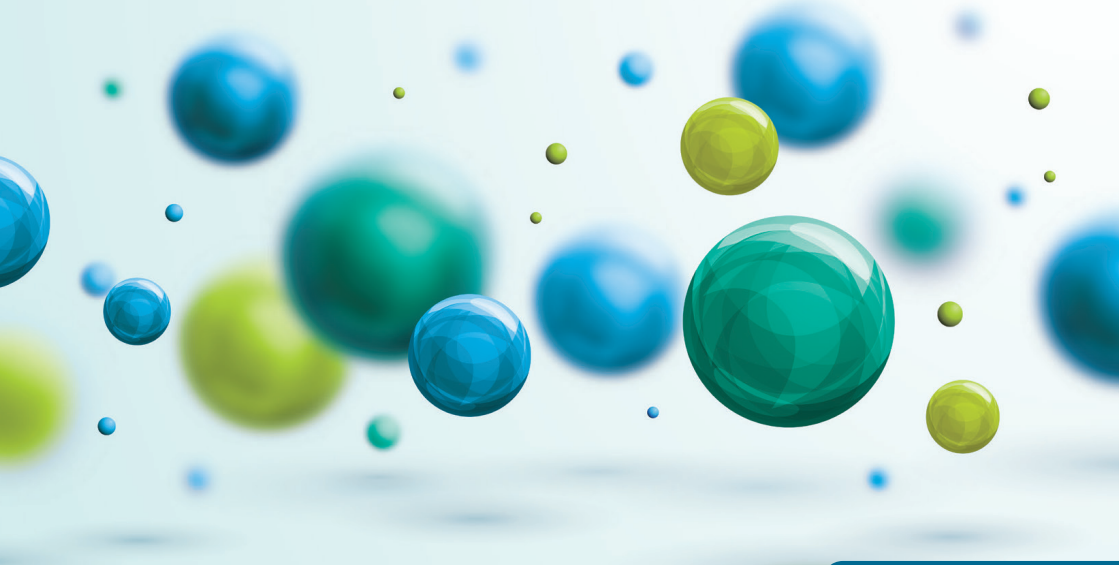

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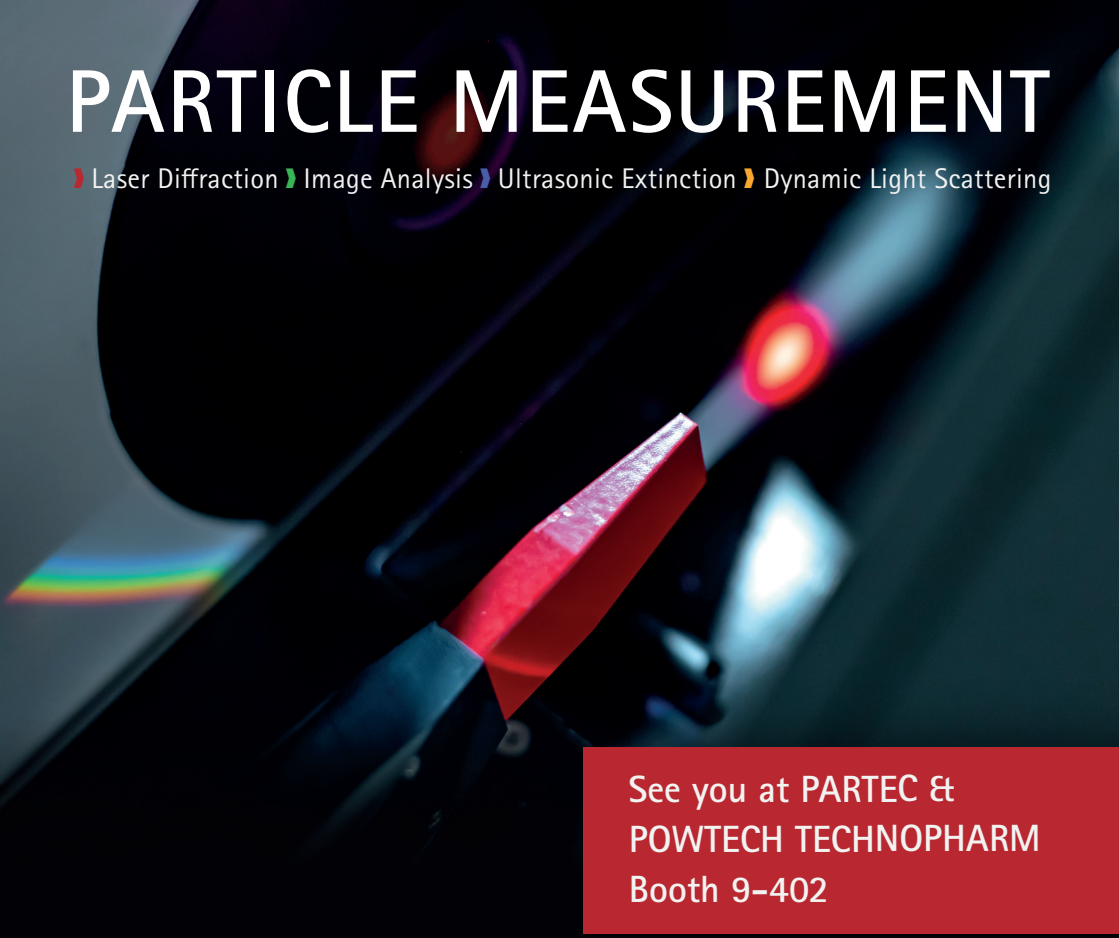

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Die Deutsche Nationalbibliothek verzeichnet diese Publikation in der Deutschen Nationalbibliographie; detaillierte bibliographische Daten sind im Internet unter www.dnb.de abrufbar.

Bibliographic information published by the Deutsche Nationalbibliothek (German National Library)
The Deutsche Nationalbibliothek lists this publication in the Deutsche Nationalbibliographie (German National Bibliography); detailed bibliographic data is available via Internet at www.dnb.de.

Der VDI-Bericht, der die Vorträge der Tagung enthält, erscheint als nichtredigierter Manuskriptdruck.
Die einzelnen Beiträge geben die auf persönlichen Erkenntnissen beruhenden Ansichten und Erfahrungen der jeweiligen Vortragenden bzw. Autoren wieder. Printed in Germany.

ISSN 0083-5560
ISBN 978-3-18-990143-6 · E-ISBN 978-3-18-990144-3

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Linking Powder Flowability Measurements to Spread Layer Quality Metrics in Additive Manufacturing via DEM

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Abstract

Predicting the quality of a spread layer in the powder spreading process for additive manufacturing methods such as Selective Laser Melting (SLM) is a rapidly growing area of academic interest. Developing easily measurable metrics for powder spreadability outside additive manufacturing machines could streamline material evaluation, optimizing spreading speed, height, and blade type for both existing and new materials without extensive in-machine testing. However, no bulk measurement has yet successfully predicted spread layer quality.

Discrete element method (DEM) simulations were used to create digital twins of a rotating drum (Granutools, GranuDrum) and a slice of the powder spreading process. A design of experiments was conducted to explore particle properties, including sliding friction coefficients, rolling friction coefficients, and cohesive energy density values. The spreading simulation further varied parameters like blade height, speed, and type, all of which influence spread layer quality.

Bulk flow measurements, such as the dynamic angle of repose and cohesive index, were recorded in the rotating drum digital twin, mirroring experimental lab setups, for all particle property combinations. Simultaneously, the spreading simulation evaluated metrics like the spreadability index and other layer quality indicators under the same particle property conditions. By linking bulk flow measurements from the rotating drum to spread layer quality metrics for specific blade types, speeds, and heights, a predictive relationship was established. This approach enables the prediction of spreadability metrics across various spreading systems using simple rotating drum measurements, offering a faster and more efficient method compared to previous approaches.

Content

Discrete differential geometric methods for machine precision simulation and parameterisation of particle-particle liquid bridges

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Abstract

State of the art models for finding the capillary forces between particles rely on semi-analytical solutions of the Young-Laplace equation. Solutions often rely on symmetries in reduced order models, the resulting forces can then be used in macroscopic simulation frameworks such as discrete element method (DEM). The difficulty lies in extending such simulations to non-symmetric particle-particle agglomerate systems where the energy minimum solution of surface interfaces can assume exotic geometries and topologies which could have radically different forces than that expected from traditional semi-analytical solutions. Building on previous work in this field which demonstrated near-exact -or machine precision- numerical solutions of minimised three-phase surface interfaces when compared to analytical solutions, we demonstrate the use of new modelling and simulations methods on more complex systems by applying advances discrete differential geometry (DDG) towards finding families of solutions of many particle three-phase interface systems.

Content

Numerical modelling and real-time magnetic resonance imaging of hydrodynamics in vibrated bubbling fluidized beds

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Prof. Dr. Stefan Heinrich
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Abstract

Mechanical vibration can improve the quality of fluidization of a system by reducing the minimum fluidization velocity and minimizing gas channeling and particle agglomeration. Despite the widespread use of vibrated fluidized beds, our fundamental physical understanding of the hydrodynamics occurring within them is still limited. The main reason for this is the fact that the spatial distribution of the phases is challenging to investigate experimentally because the systems are optically opaque. Additionally, conventional measurement techniques, such as intrusive probes, only yield solid concentration, particle velocity and bubble characteristics from one specific location of the bed and alter the flow. Therefore, non-intrusive tomographic techniques are increasingly being used to study them. Magnetic resonance imaging (MRI), a technique that has been mainly applied in the medical field, is particularly suited for obtaining spatially and temporally resolved dynamic information from the interior of vibrated fluidized beds. In addition to experimental studies, numerical simulations can be used to gain an insight into the hydrodynamics during fluidization. A promising approach for simulating fluidized beds is the method known as CFD-DEM, which combines computational fluid dynamics and the discrete element method.
In this contribution we present our progress in investigating the influence of mechanical vibrations and gas pulsation on the hydrodynamics in bubbling fluidized beds. One phenomenon that occurs at specific vibration frequencies and amplitudes is structured bubbling. It is characterized by repetitive, highly predictable bubble formations and could be observed in experiments and simulations.

Content

A Probability-Based Model for Particle Resuspension: Highlighting the Effect of Turbulence-induced Resonance

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Abstract

The phenomenon of particle resuspension plays a critical role in a wide variety of natural and industrial processes. Although being a classic problem, the particle resuspension is still not well understood yet due to the complex interplay among boundary layer fluid dynamics, article-substrate interactions, and surface heterogeneity. In this study, it is demonstrated by experiments and analysis that the turbulence-induced resonance is important for the resuspension of small particles, which is not considered in previous models. In order to properly include the contribution of the turbulence-induced resonance, we present a probability-based model for particle resuspension.
present a probabilistic dynamic model for particle resuspension in the turbulence boundary layer. Besides, in contrast to smooth surfaces, multi-scale rough surfaces may reduce the natural frequency of the particles, thereby increasing the likelihood of turbulence-induced resonance. Our model reinterprets the classic experimental measurement by Reeks and Hall and significantly improves the agreement with the experimental data. We further investigate the influence of the critical frequency ratio Ωc on resonance-driven resuspension, revealing key trends in its contribution. Additionally, within the same framework, the model is extended to unsteady flow conditions, where its predictions are validated through comparison with experimental results. Therefore, a unified model can be applied to both steady and unsteady flow scenarios.

Content

A Combined Image Analysis and Density-Based Approach to Assess the Spreadability of Metal and Polymeric Powders in Additive Manufacturing

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Abstract

In this study, it is proposed that a combination of density-based and surface quality-based approaches provides a comprehensive description of powder spreadability. Spreading tests were conducted at the University of Leeds and the University of Salerno on two polymeric and two metal powders, which were characterised using two Spreadability Indexes: the first was density-based, calculated as the ratio between powder layer's density and powder's bulk density, which had previously been applied only to metal powders; the second was surface quality-based, using image analysis and the application of a wavelet transform, which had previously been applied only to polymeric powders. In this work, the polymeric powders selected were PA6 and PPNAT, and the metal powders selected were AlSi10Mg and Ti6Al4V, all purposely produced for PBF applications. For the density-based index, mass and volume of spread powder are obtained using gravimetrical and geometrical analyses, respectively. A layer density closer to the bulk density is denoted by a higher index, reflecting better packing of the powder bed, which ensures superior mechanical properties in the final product. For the surface quality index, a high-resolution camera is employed to capture an image of the powder bed illuminated with grazing light, which is then converted into a grayscale image and divided into 1288 stripes in the spreading direction. The most significant wavelength is identified from each stripe's average wavelet power spectra. The index is defined as the ratio between the d50 of the powder and the average peak length, providing information about surface roughness. The indexes obtained at changing spreading velocity are compared and discussed for a comprehensive assessment of the quality of the spread layer.

Content

Mechanistic insights into the shape evolution and influencing factors of repose angle heaps of different dairy materials.

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Abstract

The Angle of Repose (AoR) measures powder flowability, defined as the angle between a powder heap's slope and a flat surface. However, its reliability is limited by insufficient understanding of heap formation. This study examined AoR in Skim Milk Powder (SMP), Whole Milk Powder (WMP), and micronized Whey Protein Isolate (WPI), analyzing factors like particle size, density, water content, surface fat, and restitution coefficient.
Results show powders with better flowability form heaps with smooth, normal distribution-like contours due to consistent rebound and accumulation, with the restitution coefficient playing a key role. Poorer-flowing powders exhibit frequent avalanches and irregular shapes, making AoR less reliable. For accurate flowability assessment, AoR must consider particle properties, flow states, and heap contours.

Triboelectrification of Functionalised Glass Beads during Pneumatic Conveying

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Abstract

Triboelectrification, the process of charge generation through friction, plays a crucial role in various industrial applications, including pneumatic conveying. This phenomenon can lead to significant processing issues such as particle agglomeration, equipment fouling, and even explosions. Understanding the charging behaviour of materials is essential for mitigating these risks and ensuring safe and efficient operations.

In this study, we investigate the triboelectrification of functionalised glass beads during pneumatic conveying. We address the effect of surface treatment on triboelectrification by modifying the surface properties of glass beads. This is achieved by making the beads hydrophilic through acid washing and hydrophobic through silanisation. The charging behaviour of these beads is characterised using an in-house aerodynamic dispersion method and compared with the actual charge acquired during pneumatic drying. Additionally, we study the effects of mass loading and conveying air velocity on the charging behaviour to understand how different quantities of material and air speeds influence the triboelectric charge. Experiments were conducted under both nitrogen and air atmospheres to assess the impact of different conveying environments. Our findings reveal a small but notable difference in the charging behaviour between the two environments, providing insights into the impact of functionalisation, mass loading, air velocity, and conveying conditions on triboelectrification.

Influence of bulk properties on the feeding and conveying behaviour of mineral fillers

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Abstract

The growing demand for diverse compound recipes has brought mineral fillers to the forefront of plastics compounding processes. These fillers not only reduce polymer content and lower costs but also enhance the mechanical, thermal, and rheological properties of polymer compounds. Talc and Neuburg Siliceous Earths (NSE) are particularly valued for their compatibility with a wide range of polymer matrices. However, their successful incorporation into formulations depends on precise and consistent feeding and conveying into twin-screw extruders. Despite advancements in extrusion technology, peripheral equipment such as feeders often becomes a bottleneck, limiting process reliability and product consistency.

This study highlights powder rheometry as a reliable alternative to inefficient trial-and-error methods and outdated techniques for characterising the flow behaviour of mineral fillers. Powder rheometry provides detailed, quantifiable insights into flow characteristics, enabling a more systematic approach to evaluating flow behaviour. Dynamic flow measurements, aeration/de-aeration tests, and shear cell analyses were conducted on Talc and NSE samples to assess bulk properties, including flowability, compressibility, fluidisability, and cohesion. Feeding trials using a single-screw loss-in-weight feeder evaluated dosing accuracy and consistency, with results correlated to rheological data.

The findings show that powder rheometry effectively differentiates material types, grades, and batches. Dynamic flow measurements identified material-specific differences, aeration and de-aeration tests revealed how processing conditions influence flow, and shear cell tests quantified cohesion for a deeper understanding of bulk behaviour.

Content

Coating of particles in pneumatic conveying

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Abstract

Coating is an important process in the chemical, food and pharmaceutical industry for applying flow agents, adding colorants, applying a protective layer against humidity, for the selective release of the contents or applying a flavor layer to goods. This is usually done in a fluidized bed (Wurster coater) or a coating drum. Pneumatic conveying systems are usually already installed for conveying particulate media, for example between process steps or the filling. In pneumatic conveying, the fluidized state of the Wurster Coater is already present. The basic idea of the current project is to implement the coating step directly in the pneumatic conveying system.
Experimental approach
The coating tests were carried out using a vacuum system. For this purpose, various coating chambers with different nozzle arrangements were printed using a rapid prototyper. The nozzles used were also printed in-house and adapted to the requirements.
Particles with a diameter of 3 mm were used for the tests and sprayed with a common protective coating as well as blue dye. The particles were heated to 120°C in order to supply the necessary heat to evaporate the solvent.
Images of the coated particles were taken and evaluated with ImageJ to analyze the coating result. The amount of coating delivered changes the coating layer linearly as expected. The tests showed that it is possible to coat particles during pneumatic conveying. A comparison with coating in a Wurster coater showed that the amount of overspray is higher than in a Wurster coater, leading a lower efficiency in the coating step in pneumatic conveying though.

Content

Powder evolution during successive recycling for additive manufacturing processes

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Abstract

Powder bed fusion is gathering interest in fabricating complex functional components across various industries. Reusing the remaining powder in these processes involves blending recycled material with a small portion of virgin powder to ensure consistency and performance. Product properties are evaluated through mechanical testing of standard specimens fabricated with the main components. The shortcoming is that the entire manufactured batch must be discarded in the case of unsatisfactory results, leading to waste of powder, process time, and associated costs.

This study aimed to systematically evaluate the impact of powder recycling on the physicochemical properties of particles and bulk material and they impact the mechanical properties of additively manufactured products. Optimising recycling practices is essential for enhancing the reliability, sustainability and cost-effectiveness of additive manufacturing, and producing components with desired properties.

To achieve these, multiple sets of specimens were fabricated after each round of successive recycling of PA12 nylon powder using selective laser sintering (SLS). The performance of manufactured components, and the particle and bulk properties of sample powders were evaluated using standard techniques to evaluate the changes resulted from recycling and how they correlate to the variations in the mechanical behaviour of parts.

The findings provide valuable insights into the effects of powder recycling on the mechanical properties and quality of SLS-fabricated components and identify key factors influencing performance and reliability. This research contributes to the development of more efficient powder recycling protocols, ultimately improving the feasibility and environmental impact of additive manufacturing.

Content

Multi-stage grinding for efficient recycling of electrodes: Optimizing process parameters

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iPAT

Abstract

The rising demand for electric mobility and the need for environmentally friendly recycling of lithium-ion batteries (LIBs) as well as future EU regulations requires advanced technologies to recover materials with high purity and yield.
This paper focuses on the coarse grinding and furthermore decoating of battery electrodes, which is a critical step with a significant impact on the safety and quality of the recovered materials.
Various process parameters of cutting mills, including sieve size and speed, were systematically varied and their effects on particle size, purity of the resulting black mass and yield were assessed. X-ray fluorescence (XRF) analysis was used to quantify impurities and to identify the optimum grinding conditions, with the aim of an efficient subsequent separation and recovery of valuable battery components.
The results showed that an adjustment of mill speed and precise sieve selection can increase the grinding yield without significantly increasing contamination i.e. an improvement between efficiency and material purity was explored.
A further dispersion and separation of black mass agglomerates into its individual components is challenging, particularly in view of the presence of binders, derived from the synthesis of the electrodes. These modify the properties of the components, which then interact differently compared to their primary form. The feasibility of a first separation step based on surfactants - adsorbent micellar flocculation - was investigated with the aim of removing the binder present in the mixture. These findings are crucial for the development of improved and safer recycling processes in order to increase the sustainability of battery production and reduce the dependence on primary resources.

Content

Closed loop recycling approach for aged polyamide 12 feedstock powders for additive manufacturing via precipitation combined with solvolysis

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Abstract

A major challenge in powder bed fusion (PBF) additive manufacturing of the frequently applied polyamides is the handling of already used powders (aged powder). During PBF, polyamide powders undergo chemical ageing, resulting in altered thermal and rheological properties of the feedstock material. Components made from aged powder are of inferior quality. Therefore, aged powder is usually refreshed with new (virgin) powder at a constant refresh rate. Although, a substantial amount of aged powder accumulates over the course of processing. These excess quantities are either employed in other plastic manufacturing processes (downcycling) or, more frequently, disposed, which is disadvantage from an economic perspective and in view of sustainability of the manufacturing process.
Within this contribution, a combined process of solvolysis with subsequent thermally induced liquid-liquid phase separation and polymer crystallization is presented to restore the thermal and rheological properties of aged polyamide 12 (PA12), thus making the powders suitable for PBF again. The degree of solvolysis of the aged PA12 is tuned by the reaction temperature or time. A kinetic model allows to determine suitable process parameters the process depending on the ageing condition of the feed to match the thermal properties of the recycled PA12 powders to those of PA12 virgin powder. The optimized recycled PA12 powder shows comparable zero shear viscosity, particle size distribution, particle shape and flowability to the virgin material.

Content

SiC-fiber-supported Droplet Burning for Nanoparticle Synthesis in Hele-Shaw Flow

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Abstract

Single droplet burning has been investigated extensively in combustion physics from various perspectives. Meanwhile, the disruptive burning behavior of droplets with precursors plays an important role in flame spray pyrolysis to manufacture nanopowders.
In our setup, the droplet with diameter D is introduced via a syringe with a needle and supported at the cross-section area of two crossing SiC fibers. Two glass plates sandwich the droplet to form a micro-fluidic channel, with oxidizer gas introduced and combustion products exhausted. The SiC fiber comprises smaller-diameter threads, but the radial diameter of the overall fiber is 14um. The gap distance H between the two glass plates is carefully chosen to be larger than the initial diameter of droplet D and smaller than that initial flame diameter.
Previously, the characteristic gap H of the fluid channel, flowing with gaseous air or oxygen, was much larger than the droplet diameter, presenting minimal interaction between the flame and walls. For our setup, we use a high-speed camera to track the history of droplet diameter and flame diameter, determining the instantaneous H/D ratio. As the burning droplet diameter data in confined glass plates differs from the droplet burning in an open atmosphere, we find significant interactions between the flame and glass wall when the gap H is carefully selected, as mentioned above. The walls affect the burning characteristics, and the d2-law is modified.
We have examined methanol and ethanol as fuel. The initial diameter D is about 1200um, the gap distance is 2.48mm, and the droplet burns smoothly with barely any soot. Investigations of ethanol and xylene mixtures, along with tin-based precursors, are conducted.

Content

Scalable swirl spray flame synthesis of Y_2O_3-MgO composite nano-particles for high temperature mid-infrared windows

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Abstract

Y_2O_3-MgO composite ceramics have emerged as a promising candidate for next-generation mid-infrared transparent window materials owing to the ability to maintain low infrared emittance and exhibit excellent optical and mechanical properties at high temperatures. However, a reliable and scalable method for synthesizing Y_2O_3-MgO composite particles with uniform mixing, nanoscale particle size, pure crystalline phase and high purity is still lacking, which limits the fabrication of infrared-transparent composite ceramics with superior optical and mechanical properties. In this paper, we reported the successful synthesis of pure cubic crystalline phase Y_2O_3 and MgO nanoparticles with a narrow particle size distribution and homogeneous mixing using a swirl spray flame synthesis method by optimizing precursor formulations and flame temperature. Moreover, the solid solubility limit of MgO in Y_2O_3 was investigated, revealing that flame synthesis can expand the solid solubility of MgO in Y_2O_3 to 70 mol%, which is 10 times higher than the thermodynamically stable phase diagram(~7%). Finally, infrared-transparent ceramics were fabricated using the homogeneous-mixing solid-solution nanoparticles, achieving an average transmittance of 84.9% in the 3–5 μm wavelength range at 600°C, which is close to the theoretical transmittance of 85%.

One step synthesis of TiO2-x embedded carbon nano onions via flame spray pyrolysis: A study on their morphological and optical features

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Abstract

Carbon nano-onions (CNOs) are composed of concentric graphitic layers and exhibit quantum confinement effect leading to unique morphology and wide range of optical absorption. Doping of metal oxide can significantly improve the morphology and optical features of CNOs owing to synergistic effects. In this study we carried out synthesis and systematic studies of ultrafine TiO2 embedded CNOs via flame spray pyrolysis (FSP) technique and their morphology and optical features. One step synthesis of carbon nano-anion composites was achieved using xylene and small amounts of titanium tetraisopropoxide in a closed and controlled FSP reactor. Controlled supply of oxygen through dispersion gas resulted in simultaneous formation of CNOs and TiO2 under high temperature conditions. As synthesized composite material was further processed through Soxhelt extraction to remove the organic carbon and impurities. Following this, it was pyrolyzed under nitrogen environment which leads to induce graphitic structures. Defect to graphitic nature of nanocomposite was analyzed though Raman spectroscopy and the results were further validated using OC/EC and TEM analysis. Moreover, thermogravimetric analysis was performed to investigate its graphitic behavior, and the presence of potential functional groups were analyzed with FTIR. SEM-EDx analysis confirms the presence of TiO2 (0.1 wt-% to wt-5%) in nanocomposite. Finally, UV-Vis spectroscopy analysis provides the optical absorption and band gap of the nanostructures. The results show that as synthesized nanocomposite exhibit graphitic structures with improved optical properties and for potential photocatalytic and photochemical applications.

Content

Binder distribution in continuous wet granulation using a novel small scale ring layer granulator

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Abstract

Ring layer granulation is a wet granulation process, which can be applied for the continuous production of pharmaceutical granules as an alternative to other continuous granulation techniques. In the ring layer process, a ring-shaped powder layer is formed on the wall inside the horizontally orientated process chamber by high rotational speeds of a shaft with different mixing tools attached to it. The wet granulation process is realised by continuously feeding dry powder and binder liquid into the granulator. While simultaneously conveying the wet powder to the granulator outlet, the rotating shaft exerts high shear on the ring layer, thus consolidating and shaping the granules. However, the ring layer process itself has so far been the subject of only little fundamental scientific research.
In this work, the continuous ring layer wet granulation was investigated in a novel lab scale ring layer granulator, allowing to conduct experiments with little material and energy consumption. For wet granulation, the binder distribution plays a crucial role for the final granule properties. Since process and formulation parameters influence the binder distribution as well as the ring layer process itself, the resulting granule properties were evaluated with special focus on binder distribution.
Overall, shaft speed, liquid to solid ratio and the powder feed rate showed systematic impact on the granule residence time distribution and on granule size and morphology, which in return can be correlated to the binder distribution. In general, increasing shaft speeds promote a more homogeneous binder distribution causing a more uniform size distribution and enhance granule consolidation and sphericity, while higher L/S-ratios increase the overall granule size and granulated mass fraction.

Content

Streamlining Continuous Manufacturing: Wet Granulation of High-Dose Drug Formulations Without Subsequent Drying

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BASF SE

Abstract

Wet granulation is a vital process in pharmaceuticals, traditionally requiring drying methods that can lead to high energy consumption, prolonged processing times, and significant operational costs. These conventional drying techniques often occupy considerable floor space, creating inefficiencies in high-volume manufacturing. To overcome these challenges, we present a novel wet granulation method utilizing Potassium sodium tartrate tetrahydrate (PST) as both a water donor and acceptor, effectively eliminating the need for a separate drying step.

We evaluated this innovative approach through case studies involving Metformin Hydrochloride (MET) and Paracetamol (APAP), both high-dose active pharmaceutical ingredients. The formulations were prepared by mixing MET and APAP with Fumed Silica, followed by the addition of PST and Poly(vinyl pyrrolidone) K 30. These mixtures were processed in a twin-screw extruder at controlled temperature and feed rates. The resulting granules were characterized for particle size and tableting behavior using established methods.

Our findings indicate significant particle growth attributed to the unique properties of Kollitab® PST, which releases water of crystallization at elevated temperatures, facilitating the granulation process. The granules exhibited superior flowability and compaction behavior compared to the pure APIs, meeting compendial disintegration requirements. This method not only produces high-strength tablets with satisfactory dissolution profiles but also enhances sustainability by reducing energy consumption and simplifying continuous manufacturing processes. Overall, the use of PST in wet granulation represents a significant advancement in pharmaceutical formulation technology.

Content

Understanding Fluidised Bed Melt Granulation of High-Surfactant Content Powders

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ABSTRACT

This study investigates fluidised bed melt granulation as an alternative to wet granulation for producing high-surfactant powder formulations. Using a Box-Behnken experimental design, the effects of binder particle size, binder content, and granulation time on granule properties were systematically evaluated. Polyethylene glycol (PEG) 6000 was used as a meltable binder, and granules were assessed for size distribution, integrity, dissolution, and flow characteristics. Results showed that smaller binder particles (~45 µm) produced bimodal granule distributions with improved packing and yield, while larger particles (~328 µm) enhanced mechanical strength but slowed dissolution. Medium-sized binders (~186 µm) resulted in higher attrition due to suboptimal agglomeration mechanisms. Optimal granule performance, balancing integrity and rapid dissolution, was achieved within a size range of 180–425 µm. These findings highlight the potential of melt granulation for developing fast-dissolving powder cleansers, offering advantages in energy efficiency, formulation flexibility, and product performance.

KEYWORDS

Fluidised bed melt granulation, Binder particle size, Binder content, Granulation time

1. INTRODUCTION

Powder-based cleaning products have gained renewed interest in recent years due to growing concerns around sustainability. These formulations, which typically contain high concentrations of surfactants, offer several environmental advantages, including reduced water usage, lower transport weight, and decreased reliance on plastic packaging [1, 2]. Although liquid shampoos and detergents became dominant due to their ease of processing,

packaging, and use [3], the shift toward eco-conscious consumer habits is driving a resurgence in interest for powder formats.

Amongst all powder-based cleaning products, laundry detergent powders are the most widely used and have the largest market share, however they are not as popular as liquid detergents which hold 72 % of volume share in the USA [3]. These are commonly produced using wet granulation, a process that allows for good control over particle size and structure, leading to stable, free-flowing granules [4]. However, wet granulation also has noticeable disadvantages. It typically uses water as a binder, which makes it difficult to incorporate moisture-sensitive components such as effervescent agents within formulations [5]. These components result in rapid dissolution, promote foam formation, and mimic the convenience of liquid cleaners. Wet granulation can employ organic solvents, such as ethanol or isopropanol [6], instead of water to solve this issue, however this is not considered environmentally sustainable.

Melt granulation presents an alternative to wet granulation, whereby water sensitive materials can be incorporated in powder formulations and it is suitable for high-surfactant systems. For this approach, a meltable binder replaces the aqueous one [7]. The absence of water also simplifies the drying step and reduces energy consumption. Whilst melt granulation is well-studied in areas such as controlled-release drug delivery [8], food processing [9] and fertilizer production [10], limited research exists on its use for high-surfactant content powders. To the authors knowledge, this presents a gap in understanding of how melt granulation performs in the context of dry cleansing systems.

This study presents an investigation of the impact of formulation factors on fluidised bed melt granulation, specifically binder particle size and binder content, and granulation time, on the quality of high surfactant granules produced by melt granulation. A Box-Behnken experimental design was used to study the impact of these parameters on granule size, integrity, dissolubility, compressibility, and bulk density. This approach would allow for the industrial implementation of a low shear granulation approach with non-aqueous binders, under a range of formulation conditions.

2. EXPERIMENTAL METHODS

Preparation of powders

Flakes of polyethylene glycol 6000 (PEG6k) (Merck, Dorset, UK), a hydrophilic meltable binder, were milled with a Retsch Planetary Ball Mill PM 100 (Retsch, Leeds, UK) and sieved into three separate sizes of ~45 µm, ~188 µm and ~328 µm. Sodium dodecyl sulphate (SDS) (Merck, Dorset, UK), a non-ionic surfactant, was used as the model surfactant. Citric acid and sodium bicarbonate (Merck, Dorset, UK) was used as the effervescent component. Microcrystalline cellulose (MCC), grade 112 was used as the excipient (Fisher Scientific UK, Leicestershire, UK). The amount of material used was fixed for all batches except for PEG6k which varies between three levels, as shown in Table 1.

Table 1. Inclusion level of each material.

Material	Amount (g)	Inclusion Level (%)
PEG6k	90/107/123	18-23.1
Sodium dodecyl sulphate (SDS)	82	15.4-16.4
Microcrystalline cellulose, grade 112 (MCC)	205	38.5-41.0
Citric acid (CA)	61.5	11.5-12.3
Sodium bicarbonate (SB)	61.5	11.5-12.3

Granule production

Granules were produced using a 3 L fluid bed granulator (Glatt GPCG 2, GmbH, Germany). An in-situ melt granulation method was utilised, involving three steps: heating, granulation, and cooling. In the heating phase, all components, including the binder, were loaded and heated with air at a flow rate of 20 Nm³/h at 113 °C, the superficial velocity is 0.68 m/s ~1.36 U_{mf} (minimum fluidisation velocity), until the product temperature rose from 25 °C to 60 °C. The granulation phase then began, with continued heating for a specific duration, resulting in a final product temperature between 60 °C and 72 °C. Finally, the cooling phase involved applying

air also at a flow rate of 20 Nm³/h at 33 °C to reduce the product temperature to 40 °C, at which point the process was stopped, and the granules were collected for subsequent analysis.

The investigated variables were binder particle size (X_1), binder content (X_2), and granulation time (X_3). The experiments were performed in a randomised order according to the Box–Behnken design, giving a total of 15 experiments. Real and coded values of the investigated variables are given in Table 2. Analysis of variance (with a confidence interval of 0.05) was performed to determine the significance of each factor and a response surface methodology was used to show the quadratic interaction between the factors where applicable. The response variables were yield, coarse level, spouted bed test and solubility test.

Table 1. Real and coded values of evaluated factors

Independent variable	Symbol	Coded and real values		
		(-1)	0	(+1)
Binder particle size (µm)	X_1	45	187.5	337.5
Binder content (% wt.)	X_2	18.0	20.7	23.1
Granulation time (min)	X_3	3	7.5	12

Granule characterisation

Particle morphology and size analysis

Morphological analysis of raw materials and produced agglomerates was conducted using a Hitachi S3400-N SEM, with samples mounted on carbon tape for micrograph acquisition. The particle size distribution of the raw materials was determined via laser diffraction (Sympatec Helos, GmbH, Germany) [11]. The final product from the fluid bed was sieved into separate fractions (Retsch GmbH, 180-4000 µm range) [12], and the yield was calculated as the weight percentage of granules in the size range 180-1400 µm, similarly the coarse level and fines level was calculated as the weight percentage of granules in the size range > 1400 µm and < 180 µm, respectively.

Attrition resistance

The spouted bed attrition test assesses the breakdown of granular materials into finer fragments under mechanical stress, a crucial factor in various industries. This method, conceptually similar to pneumatic transport attrition tests, evaluates particle breakage in conditions relevant to fluidised transport systems [13], unlike impact-based methods [14]. The test involves fluidising a 25 g sample of pre-sieved granules (180-1400 µm) in a spouted bed apparatus with controlled airflow (0.49 m/s or 14.5 L/min, ~0.6 bar) for five minutes under controlled environmental conditions (25 ℃, 50% relative humidity). After the test, the sample, including collected fines, is re-sieved at 180 µm, and attrition is quantified as the percentage increase in fines. This provides insight into material durability during processing, storage, and transport.

Dissolution testing

The T_{90} dissolution test quantifies the dissolution rate of granules by continuously measuring changes in solution conductivity using a Metrohm 856 Conductivity Module with Tiamo software. This method, relevant to cleaning formulations and drawing parallels to pharmaceutical dissolution testing, assesses ionic release as a proxy for dissolution kinetics [15]. The test involves adding 1 g of granules to 600 mL of demineralized water in a temperature-controlled (25 ℃) 1-litre jacketed vessel with a magnetic stirrer (vortex depth ~40 mm). Conductivity and temperature are continuously monitored using a combined probe calibrated with KCl standards. The test runs for a predefined period (500-600 seconds), and the dissolution rate is analysed by determining the T_{90} value from the conductivity vs. time curve, representing the time required to reach 90% of the final conductivity. This provides a quantitative assessment of how quickly and completely the granules dissolve.

3. RESULTS & DISCUSSION

Particle Properties (Morphology, Size, Distribution)

SEM micrographs of the primary materials used in the formulation, together with the values of volume mean diameter (VMD obtained by laser diffraction (Sympatec Helos, GmbH, Germany) are given in Figure 1. The morphologies are markedly different, with the SDS particles being generally spherical (Fig. 1A), the MCC fibrous (Fig. 1B) and the CA and SB being crystalline (Figs. 1C, D). The VMD of the MCC is approximately 40% that of the other materials, however SDS possesses a significantly lower density, resulting in the mixture

demonstrating a dusty nature, a key driver for granulating this system. The elongated shapes of MCC and SB and their small sizes can hinder powder flow due to higher surface area for interparticle interactions [16, 17].

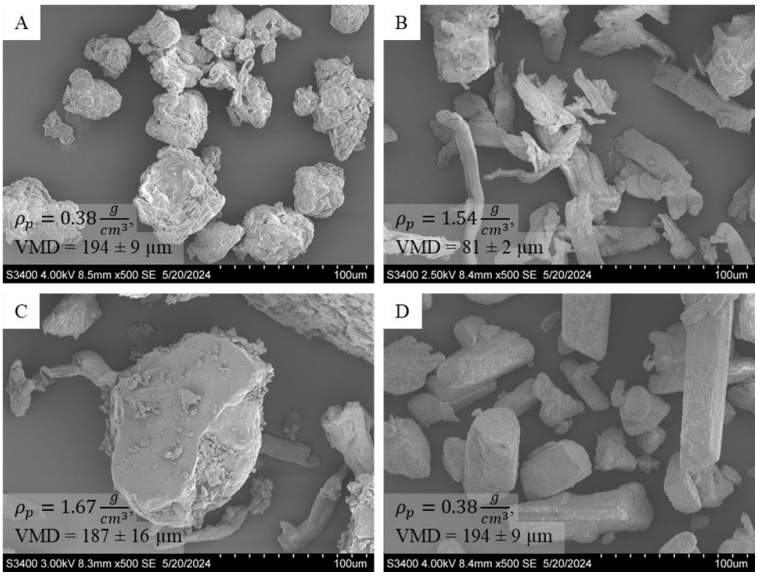

Figure 1. Scanning electron microscopy micrographs of primary particles including the particle density $\rho_p(\mathrm{g/cm^3})$ and the VMD of each material. (A) SDS, (B) MCC, (C) CA, (D) SB

Figure 2. shows the SEM micrographs of the fines (A; < 180 µm) and the granules (B) at the end of the process. The fines can be seen to predominantly consist of MCC which is expected as it is the component with the highest inclusion level which has irregular size and shape (Fig 2A); in contrast, the granules produced have a more regular size and spherical shape (Fig 2B). These spherical granules are highly desirable due to their enhanced flowability when compared with elongated or irregular particles because their shape reduces interparticle friction [18], minimises cohesive forces [19], and promotes uniform packing with consistent void spaces [20], all of which facilitate smoother and more efficient movement.

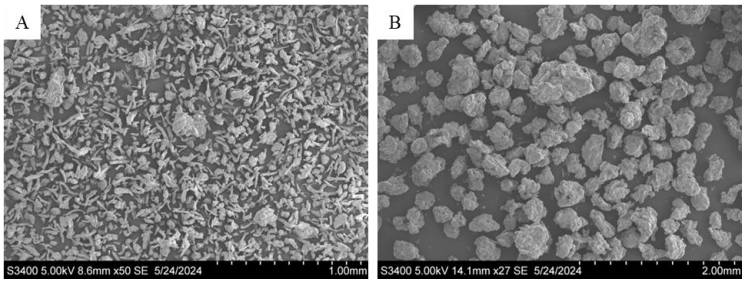

Figure 2. SEM micrographs of the final product. (A) Fines (< 180 μm) and (B) Granules (180-1400 μm)

Figure 3. shows the granule distribution density, plotted as the function of the sieve aperture size as shown in Eq. (1). for granules produced from three different binder particle sizes. When small binder particles were used, the distribution is bimodal, with peaks around 200 μm and 600 μm. This increases the packing efficiency since smaller particles fill the voids between larger ones, leading to denser and more stable compacts [21]. In contrast, medium binder particles (~186 μm) results in a monomodal distribution peaked around 500 μm, suggesting a more uniform but less efficiently packed structure. Larger binder particles (~328 μm) are also monomodal, though slightly broader, indicating even coarser granules. Smaller binder particles are more favourable due to the better packing characteristics of the bimodal distribution.

$$f(log(x)) = \frac{w_i}{log(x)_i - log(x)_{i-1}} \quad (1)$$

where x is the aperture size (μm), w_i $(\%)$ is the mass fraction in the ith interval and x_i (μm) is the top size of interval i.

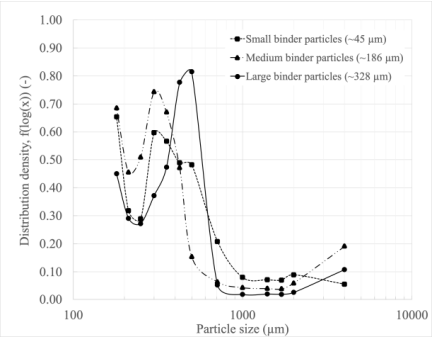

Figure 3. Granule size distribution (GSD) curves for the granules produced using small (~45 µm), medium (~186 µm), and large (~328 µm) binder particle sizes.

Figure 4. illustrates the response surfaces for yield (180-1400 µm) and coarse level (> 1400 µm) as functions of binder particle size and binder content, the only two variables significantly affecting both responses. Binder content exhibits a linear influence, while binder particle size shows particle size shows both linear and quadratic effects. The yield surface (Fig 4A) suggests that the maximum yield lies outside the experimental design space, as indicated by the curvature of the response surface, implying that the optimal conditions were not captured within the tested range. The highest yield was achieved using the smallest binder particle size and the lowest binder content. Similarly, the coarse level surface (Fig 4B) reaches a minimum under these same conditions, indicating that higher binder contents led to over-agglomeration. Given the target yield of 70 %, minimising coarse fraction is critical, as excessive agglomeration reduces the proportion of granules within the desired size range.

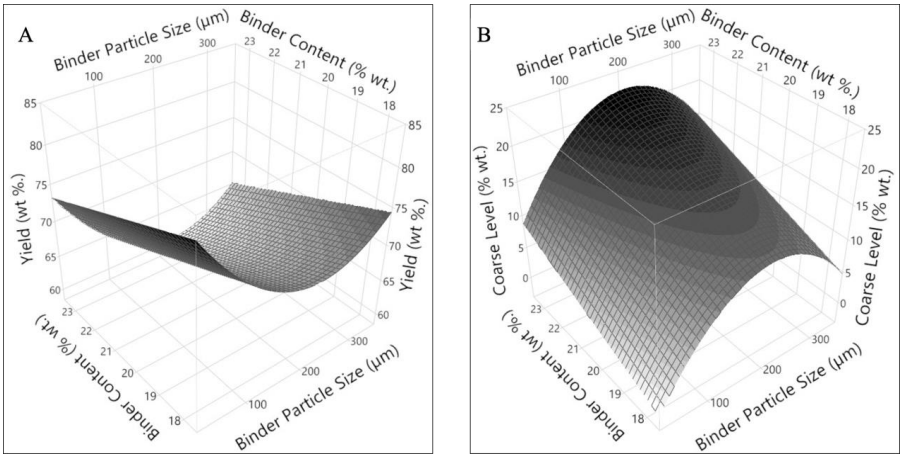

Figure 4. Response surfaces for the effect of binder particle size and binder content on yield (A) and coarse level (B).

Granule Integrity (Attrition Resistance)

The results from the spouted bed test indicate substantial breakage during testing, with fines (< 180 µm) increasing by 32-39 % and the relationship between binder particle size and attrition follows a quadratic trend, shown in Figure 5. Initially, increasing binder particle size increases the percentage of fines generated, but beyond ~200 µm, further increases lead to lower granule attrition. This behaviour is also seen in [22] where an increase in binder particle size led to a marked increase in granule integrity from binder particle size of < 250 µm to 710-1000 µm. This could be explained by the agglomerate formation mechanisms [8], whereby granules formed with medium binder particle sizes break down more because they lack the structural integrity seen with larger binders and do not benefit from the uniform binder distribution achieved with smaller particles. Medium-sized binders fall between the immersion and distribution mechanisms, where neither immersion nor distribution mechanisms dominate effectively, leading to less stable granule structures.

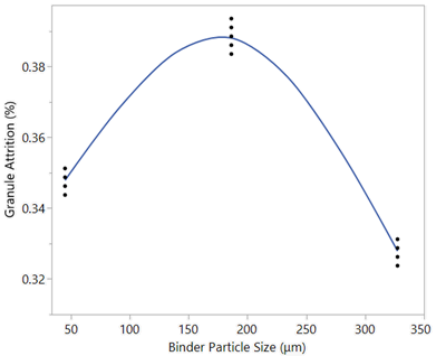

Figure 5. Effect of binder particle size on granule attrition.

Dissolution Performance

Figure 6. shows that granule dissolution time (T_{90}) increases with particle size, with the 180-425 µm range offering the most favourable balance, dissolving in approximately 5 seconds. Granules within this size range benefit from the fluidised bed process, which applies low shear to create a more porous structure, enhancing water penetration and dissolution, compared to the denser melt granules formed through high-shear methods [23]. As previously discussed, using larger binder particles can improve granule strength by forming more robust agglomerates; however, this also results in larger granules with slower dissolution, which is undesirable. Therefore, a balance must be struck between achieving sufficient mechanical integrity and maintaining fast dissolution.

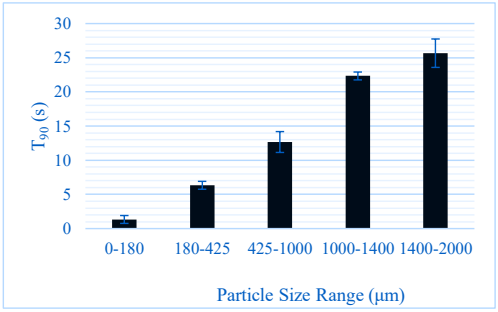

Figure 6. Dissolution time (T_{90}) of granules of different size ranges.

5. CONCLUSIONS

This study demonstrates the feasibility and effectiveness of fluidised bed melt granulation for granulating high-surfactant content powder formulations, offering a water-free alternative to traditional wet granulation. By systematically evaluating the effects of binder particle size, binder content and granulation time using a Box-Behnken design, the research provides valuable insights into how these parameters influence granule quality. Small binder particles (~45 µm) produced bimodal granule size distributions, which enhanced packing efficiency and yield, while minimising the formation of oversized granules. In contrast, larger binder particles led to more uniform but coarser granules, with reduced fines and improved mechanical integrity. However, medium-sized binders (~186 µm) resulted in the highest attrition, likely due to suboptimal binder distribution and incomplete agglomeration mechanisms. Dissolution testing revealed that granules in the 180-425 µm range dissolved most rapidly ($T_{90} \approx 5\ \mathrm{s}$); balancing mechanical strength and dissolution performance will be crucial going forward. The study also highlighted the excessive binder content can lead to over-agglomeration, reducing yield and increasing coarse fractions. Overall, the findings support the use of fluidised bed melt granulation low-shear method for producing dry cleansing systems with tailored performance characteristics. The ability to fine-tune granule properties through formulation and process parameters opens new opportunities for environmentally friendly, high-performance powder products.

LIST OF SYMBOLS

X_1	Binder particle size	[µm]
X_2	Binder content	[% wt.]
X_3	Granulation time	[min]
$f(\log(x))$	Distribution density function for granule size	[-]
w_i	Mass fraction in the i-th sieve interval	[µm]
x	sieve aperture size	[µm]
x_i	Top size of the i-th sieve interval	[µm]

ACKNOWLEDGEMENTS

The authors acknowledge the financial support provided the Engineering and Physical Sciences Research Council (EPSRC) through the Centre for Doctoral Training in Formulation Engineering (grant number EP/L015153/1) and including contributions from Unilever Ltd.

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Hierarchical Iron-Oxide Supraparticles: A High-Efficiency Adsorbent for Water Pollution Remediation

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Abstract

Water pollution caused by toxic heavy metals and organic dyes poses a significant threat to ecosystems and human health due to their persistence and toxicity. Water treatment methods like adsorption are essential due to their simplicity, cost-effectiveness, and high efficiency. Iron-oxide nanoparticles are effective adsorbents due to their superparamagnetic properties and ease of functionalization. Nonetheless, the small volume of iron-oxide nanoparticles makes their recovery after adsorption within short periods challenging. Herein, we present a scalable method to synthesize superparamagnetic iron-oxide supraparticles for efficient adsorption and removal of heavy metal ions and dyes. These supraparticles were fabricated using a spray-drying process, resulting in stable, porous structures that enhance the magnetic properties of the nanoparticles.

The iron-oxide supraparticles exhibited excellent removal efficiencies for Pb(II), Cr(III), Cd(II), Cu(II), Ni(II), Co(II), Li(I), and methylene blue, with maximum adsorption capacities of 500, 446, 417, 366, 315, 294, 286, and 670 mg/g, respectively, ranking them among the top adsorbents. They also demonstrated resistance to co-existing ions and natural organic matter, indicating their suitability for real-world wastewater treatment. Post-adsorption structural analysis revealed mechanisms for their superior performance.

The hierarchical structure enhances adsorption by providing accessible active sites for pollutants. Additionally, it improves magnetic properties for efficient recovery within seconds via magnetic separation. This architecture ensures mechanical stability and reusability, maintaining performance across multiple cycles, representing a significant advancement in developing sustainable water pollution remediation.

Content

Investigation on removal of ionomer from PEMEL catalyst particles and the impact on mechanical separation process based on their (de)wetting ability

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Helmholtz-Zentrum Dresden-Rossendorf e.V.

Abstract

The requirement of energy transition for the decarbonization is leading the investigation on water electrolysis system, which can produce high purity of green hydrogen with renewable energy resources. As various critical raw materials such as platinum group metals (Pt, Rd, Ir, and Rh) are used in proton exchange membrane water electrolyzers (PEMELs) as catalysts, ongoing studies focuses on developing their recycling processes. Because ionomer (e.g. Nafion, a perfluorosulfonic acid polymer) is added to the electrode particles to act as a binder and proton transfer agent, conventional recycling processes can release toxic hydrogen fluoride (HF) gas as a decomposition product as well as microscopic PFAS.
This study introduces the methodologies to remove ionomer without creating significant safety hazards during the separation processes. Solvents, plasma treatment, and microwave treatment can be proposed to remove the Nafion from the catalyst particles. Surface characterization of the particles is necessary because the effect of the amphiphilic molecule (Nafion) on the wetting behavior of the particles has not been clearly investigated. Furthermore, the influence of removal process on the separation processes of anode and cathode particles is identified with particle separation methods. For the investigation, Carbon Black (CB) and titanium dioxide (TiO_2) are used as representative materials for platinum-containing CB and IrO_2 catalyst particles, respectively. The removal of the ionomer worked partially with the investigated methods, and it leads the change in surface characteristics of the particulates. We provide the possible sustainable path of recycling processes of the PEMEL membrane electrode assembly unit, and point the way to the next research steps.

Content

Fine particle flotation for recycling black mass from lithium iron phosphate batteries

Aliza Salces[1]; Chiedza Nzuma[2]; Dr. Duong Hoang[1]; Prof. Dr. Anna Vanderbruggen[3]; Dr. Martin Rudolph[1]

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Abstract

Froth flotation presents a promising technique for recovering valuable components from the black mass, i.e., fine fraction generated during the comminution/crushing process, of lithium iron phosphate (LFP)-type batteries. However, the ultrafine nature of LFP particles and their carbon coating pose significant challenges. This study investigates the flotation behavior of LFP black mass using pristine spherical graphite and LFP particles and a model LFP black mass (MBM) comprising a 1:1 mass ratio. Industrially pyrolyzed LFP black masses (IBM) were subsequently tested. Flotation experiments were conducted using a conventional mechanical cell and a pneumatic flotation cell which is suited for fine particle flotation. Flotation of single-components using the mechanical cell resulted to recoveries of 5.1 % for pristine LFP and 93.3 % for pristine graphite in the overflow (O/F) product, indicating potential for separation. However, flotation of MBM demonstrated limited graphite grade and recovery of 38.9 % C and 45.5 %, respectively. In MBM, a sliming effect of the ultra fine LFP particles lead to this low graphite recovery and grade. In contrast, flotation of IBM achieved a graphite grade and recovery of 77.7 % C and 84.5 %, respectively. Pneumatic flotation of IBM exhibited comparable performance to mechanical flotation, achieving a graphite grade and recovery of 79.6 % C and 83.0 % in recirculation mode and 77.6 % C and 89.0 % in one-pass mode.

Recovery by entrainment remains the predominant factor for fine LFP recovery. The coarser particle size of LFP in IBM reduced this entrainment. We will have a deepened discussion on the fundamental separation mechanisms and a comparison of the materials and the separation technology tested.

Content

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- **Gas Adsorption (BET)** – Surface area and porosity
- **Gas Pycnometry** – True and envelope density

Global analysis of multiwavelength analytical ultracentrifugation data sets from sedimentation velocity experiments

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Abstract

To determine the structure-property relationships of particle ensembles it is crucial to know their size, shape, optical properties and their distributions. Methods like microscopy or scattering techniques are often limited by statistics or resolution for broad distributions. Analytical ultracentrifugation (AUC) overcomes these drawbacks by coupling gravitational separation of particles with spectroscopic detection, allowing high-resolution analysis of broad distributions. Furthermore, multiwavelength (MW) AUC provides the optical extinction or emission properties together with the hydrodynamic properties, resulting in detailed particle size distributions along with their optical properties. However, so far MW analysis using direct boundary models was challenging due to its high computational cost.
To overcome these limitations, we developed the tool HDR-SVFIT that enables the rapid, simultaneous and global analysis of MW-AUC datasets from sedimentation velocity experiments for all measured wavelengths. It enables regularisation in both, sedimentation coefficient and wavelength dimension, addressing the issue of distribution broadening. Besides that, optical spectra of individual species can be extracted with high resolution or spectral trends can be determined.
Our approach enhances the application of MW-AUC for multidimensional analysis of particle systems, providing insight into their size and shape distributions and related spectra. We will present its effectiveness in characterising complex colloids, like plasmonic or semiconducting nanoparticles and bionanoparticles. Since knowing the correlation between size, shape and optical properties is essential for understanding the structure-property relationships our methodology is an important contribution to this field.

Content

Control of Buckling of Colloidal Supraparticles

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Abstract

Clusters of colloidal particles, often termed supraparticles, can provide more functionality than the individual particles they consist of. Since these functionalities relate to the arrangement of the primary particles within a supraparticle, understanding and controlling the structure formation process is an important topic. The consolidation of the particles into crystals can be prevented if the structure buckles. In this study, we show how to control buckling of colloidal supraparticles in a water/oil emulsion produced by microfluidic techniques. We control the degree of buckling by tailoring the interaction of the negatively charged particles with the surfactants at the water/oil-interface. More specifically, a mixture of non-ionic and anionic surfactants stabilizes the emulsion. We use a local change in pH in the aqueous phase to control the particle-surfactant interactions, by inducing the protonation of the anionic headgroup. After protonation of the anionic headgroup, the particles can adhere to the w/o interface. Further drying leads to increased pressure on the formed shell and buckling of the structure. We show that the pH of the aqueous phase changes during the assembly and by controlling the initial pH, we gain full control of the final morphology ranging from thin-sheet like buckled structures to regularly undulated morphologies to spherically rough and finally spherically smooth structures. The ratio of particles at the interface to bulk during the protonation step will determine the final structure. A high ratio leads to strongly buckled structures while low ratios result in more spherical clusters.

The effect of seed variation on the as-synthesized and aged morphology and optical properties of silver patchy particles

Monika Stadelmaier; Prof. Dr. Robin Klupp Taylor
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Abstract

Noble metal nanoparticles have gained interest due to their localized surface plasmon resonance (LSPR), which enables their use in a wide range of applications such as theranostics, pigments, sensing, and photocatalysis. The nanoparticle optical properties are influenced by their size, shape, and composition. Thus, in particular anisotropic nanostructures like patchy particles, where thin noble metal patches partially cover spherical dielectric cores, offer high tunability. Our research group developed a continuous flow setup to scalably produce silver patches on charged core particles, involving low-level seeding and electroless deposition of silver ions. Through adjustment of the silver precursor-to-seed ratio, patchy particles with a tunable LSPR between 400 and 1300 nm wavelength can be produced [Völkl et al., ACS Appl. Nano Mat. 2023].
This contribution details our investigations into how seed age and morphology influences the shape and LSPR of patchy particles. We observed that the LSPR peak shifts if seeded core particles are aged. Besides that, our study systematically varies seed size and shape and employs scanning electron microscopy and high-angle annular dark field scanning transmission electron microscopy to relate the final patch morphology and optical properties. Moreover, we investigate the long-term stability of patchy particles, revealing the effects of silver oxidation and silica dissolution during storage in certain conditions, while sucrose stabilization mitigates silver oxidation and helps preserve the LSPR peak position. Understanding these aspects will lead to optimized processing and storage conditions for seeded core particles and grown patchy particles, a key step towards exploiting these tailored nanostructures in various applications.

Content

Silica Particles with hierarchical porosity

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Abstract

Porous materials are essential for several technical applications, such as filtration, catalysis supports, scaffolds for biomedical applications, stationary phase materials of chromatographic columns.
Porous glasses, for instance, can be manufactured by melting borosilicate glasses that phase separate at high temperatures. One of the phases is susceptible to acid, and thus can be etched away, leaving behind a porous structure. Micrometer sized objects or morphologies are hard to achieve with this type of technology. On the other hand, a self-assembly approach can create tailored supraparticles from the confined agglomeration of uniform silica or polystyrene primary particles. The porosity of such materials can be templated by the size of the primary particles. Without further processing, the material will display mainly one range of pores: macro, meso or micropores.
However, many of the applications that require porosity would benefit from a material with bimodal or hierarchical porosity. Macropores increase mass transport and thus efficiency and throughput and reduce pressure drop, while micro and mesopores simultaneously provide sufficient surface area and active sites.
Here, we discuss approaches on how to create particle systems with tailored, hierarchical porosity. Our method allows us to tailor particle size and particle size distribution and tune the size of both macro and mesopores independently from one another, to suit the application of choice.

Content

Size and composition controlled synthesis of bimetallic silver-gold alloy nanoparticles

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Institute of Particle Technology – Friedrich-Alexander Universität Erlangen-Nürnberg

Abstract

Plasmonic nanoparticles with a core size below 100 nm show interesting electrical, catalytic and especially optical properties. Due to these properties, plasmonic NPs find application in a variety of research areas, including optics and nanocatalysis. Silver (Ag) and gold (Au) are the benchmark materials for strong plasmon resonances in the visible spectrum, thus AgAu alloy NPs are of special interest, especially for optical applications. Using AgAu alloy NPs in real world applications requires precise control over their size, shape and chemical composition. Due to the complex formation mechanism of bimetallic NPs, changes in one of those properties often result in an uncontrolled shift in the other.
In this work, we demonstrate how the reaction pathway of AgAu alloy NPs can be tailored to enable independent control over the size and the chemical composition of the produced NPs. NP synthesis involves control of the supersaturation profile in time and space. For the reaction-controlled case, this supersaturation profile is determined by a network of reactions leading to the buildup of the monomer concentration.
The synthesized NPs are analyzed using a set of advanced characterization tools. Specifically, size exclusion chromatography is used to enable the characterization of a large number of samples in terms of size and chemical composition.
Current research goes towards the complete exclusion of human experimenters via automated synthesis platforms. Doing so will not only increase the reproducibility with regard to size, shape and chemical composition across different experiments but also enable high throughput experimentation.
Funding of the German Science Foundation (DFG) through the collaborative research center (CRC) 1411 is gratefully acknowledged.

Content

An Automated Calibration Tool for Uncertainty Quantification in DEM Simulations

Giulia Tranquillini[1]; Paul Kieckhefen[2]; Lukas Kropfinger[2]; Philipp Grohn[3]; Prof. Johannes G. Khinast[1]; Dr. Thomas Forgber[1]
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Abstract

Discrete Element Method (DEM) simulations are essential for studying granular flow and powder mechanics in research and industry. These rely on contact models governed by microscopic parameters, which significantly influence macroscopic material behavior. However, calibrating these parameters to match experimental bulk properties remains challenging, often requiring time-intensive, manual adjustments or optimization algorithms prone to human bias.

We present an automated calibration tool to streamline this process and reduce subjectivity. Built using a dataset of thousands of artificial powders characterized by Luding model parameters and bulk properties, the tool allows users to input observed powder behavior and receive optimized contact parameters. It enhances calibration accuracy and efficiency by identifying parameter sets that best match the input data.

A notable feature is its ability to handle incomplete datasets. When bulk data is missing or unreliable, the tool identifies powders with similar behavior and highlights those with the greatest variation in missing tests. This guides test selection for expensive or hazardous materials, prioritizing measurements that most effectively reduce uncertainty. By quantifying variance across similar powders, the tool provides a direct measure of uncertainty in simulation results.

To validate the tool, we tested it on limestone powder processed in a small-scale feeder. Performance was assessed by comparing parameter sets under scenarios with incomplete data. Variations in simulation outcomes quantified uncertainty, and numerical results were validated against experimental data, confirming the tool's reliability and value for DEM calibration.

Content

CFD-DEM analysis of droplet drying and solidification in spray drying processes

Dr. Moritz Buchholz[1]; Riccardo Togni PhD[2]; Dr. Dominik Weis[1]; Dr. Christoph Goniva[2]; Prof. Dr. Stefan Heinrich[3]
[1] BASF SE; [2] DCS Computing GmbH; [3] TU Hamburg

Abstract

The spray drying process enables the efficient production of large quantities of granular material from liquid solutions or suspensions. This process is typically a step in a larger production chain, which includes upstream process operations such as synthesis, and downstream processes for additional drying, sieving, or further processing of the granular material. In addition to their size distribution, the morphology of the spray-dried particles has a significant influence on the resulting quality and handling of the product material. For example, hollow particles are characterized by lower tensile strength, which can lead to an increased fragmentation of the product. The particle morphology can be influenced by the drying conditions experienced in the spray dryer, as well as by the upstream processes that determine the properties of the drying material. Due to the challenges of scaling-up the results obtained from laboratory experiments to larger scales, computer simulations can be used to support the design and optimization of facilities at production scale. This contribution presents a novel unresolved CFD-DEM simulation approach for modelling single suspension droplets undergoing drying and a solidification process in a hot gas. The key mechanisms of the model, which describe the particle-fluid interactions, are the energy and mass exchanges with the surrounding gas. The droplet solidification is accounted for by the formation of solid bonds between the primary particles in the suspension. A sensitivity analysis reveals that the properties of the solid bonds determine the droplet´s drying rate and can thus lead to the formation of particles with different morphology.

Content

Multilayer Recoating and Flexible Blade Dynamics in Metal AM: A DEM Approach

Dr. Gustav Kettil PhD; Dr. Johannes Quist PhD; Dr. Negar Sani PhD; Prof. Fredrik Edelvik PhD
Fraunhofer-Chalmers Centre

Abstract

Metal additive manufacturing (AM) has revolutionized industrial production, enabling complex and customized components. A critical step in powder-based AM processes is the recoating process, where a layer of powder is evenly spread over the build area. The quality of this step significantly impacts the uniformity and integrity of the final product. Flexible blades are often used in recoating to ensure consistent powder spreading, as they can conform to surface irregularities and reduce the risk of damaging previously built layers. AM is a multilayer process, and each new layer interacts with the surface left by the previous one, where defects can accumulate and propagate. Understanding this multilayer nature is essential for optimizing recoating strategies and ensuring consistent, high-quality builds. Discrete Element Method (DEM) simulations provide insights into the particle dynamics during recoating, helping to understand and mitigate issues like uneven layer thickness and particle segregation. Incorporating flexible blades and the multilayer nature into simulations, however, introduces significant complexities. In this work, we developed a coupled framework with a DEM solver and a finite element method solver to simulate flexible blade deformation during interactions with powder and the build platform. Additionally, we implemented a solidification approach to enable simulations of multiple recoating passes, capturing the cumulative multilayer effects of the multilayer process. We present the challenges encountered and the key findings from this study, demonstrating advancements in the predictive capabilities of DEM simulations. This work contributes to the optimization of recoating strategies, ultimately improving part quality and process efficiency in metal AM.

Content

Efficient DEM simulations of polarizable particles including tribocharging and electrostatic interactions

Maria Giordano; Dr. Francesca Orsola Alfano PhD; Dr. Giovanni Iozzi; Francesco Paolo Di Maio; Prof. Alberto Di Renzo
University of Calabria (UNICAL)

Abstract

Electrostatic charge exchange during particle-particle and particle-wall collisions can result in charge build-up, particularly in dielectric materials. This phenomenon affects industrial processes by causing irregular flow, segregation, and particle adhesion, ultimately compromising product quality. Particle-scale simulations provide valuable insights into the dynamics of charge transfer and accumulation, as well as their effects on particle motion. However, fundamental aspects of tribocharging mechanisms, including the role of surface charge heterogeneity in interactions between polarisable particles, remain debated and current modeling approaches appear insufficient to capture the full range of behaviors observed experimentally.

We present a charge-plus-effective-dipole model as a viable solution for particle-scale simulations involving large numbers of charged particles. Adapted from a literature model of polarisable ions to solid particles, the approach accurately captures realistic attraction forces between like-charged particles and between charged and neutral particles, aligning with rigorous solutions. Implemented within a Discrete Element Method (DEM) framework, this model enables the efficient simulation of large-scale systems comprising hundreds of thousands of interacting particles, revealing how polarisation effects influence charged particle motion and interactions during shaking. Then, surface polarisation in the context of charge transfer (tribocharging) process is discussed, particularly in relation to charge difference amplification.

Content

Towards a true measurement of powder rheology

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Abstract

Many powder processes operate under dynamic conditions, such as blending and mixing. To optimise these processes, it is beneficial to understand the prevailing shear stresses in the powder bed under these high strain rate conditions. Measurements of powder shear stress under quasi-static conditions is well established using shear cells. However, direct measurement of shear stress at elevated strain rates is limited to powder couette devices, which are not commercially available and are difficult to operate particularly for fine cohesive powders. Commercial instruments for dynamic powder flow assessment are limited to dynamic angle of repose variation and torque or energy input in powder rheometers. In this work we simulate the FT4 Powder Rheometer using the Discrete Element Method (DEM) and show a robust relationship between the prevailing shear stress and the impeller torque and volume of the mobilised powder bed. Rheometer measurements using several granular materials, coupled with high-speed camera measurements to establish the mobilised bed volume, enable the shear stress to be predicted from the above relationship. By measuring in the quasi-static regime and comparing to shear cell results, we show that the above relationship has promise. This approach enables the shear stress required for flow to be determined as a function of strain rate using a commercial rheometer, enabling true characterisation of powder rheology.

And make measurable that which is not: How to deal with terrible samples in powder flow measurements

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Abstract

Both pre-shear/shear cells as well as stirrer-based measurements have become ubiquitous in recent decades and have more or less become standardized across industries and fields. This does not however mean that they are easy to use on anything besides cooperative samples. While what constitutes a cooperative sample is different from method to method problems with unusual powders with unusual properties persist.
While the basis of powder mechanics in the silo case or storage is well understood, a shear cell will be hard pressed to produce the necessary values to apply them even with relatively common cases such as fibers, powders with unusually high air retention, fast time consolidation, partially fibrous samples or even with something as common as infant formula.
This talk will take the form as a series of industrial case studies on one hand with both a defined industrial problem and solutions that may take the form on measurement schemes and adaptations to the classical measurement modes in order to generate both process insight as well as usable data for process monitoring and quality control.
The more theoretical side will analyze the possible solutions in regards to the practice of calibration, namely how especially in shear cells over und underconsolidation, often utilized to make samples more cooperative, influence not only the resulting values but the preconditions as well, therefore skew calibration towards lower or higher values of c, sigma c and sigma tau.
To make it less depressing the talk will conclude with an application of these measurement modes to a more cooperative sample made uncooperative by sintering it inside a shear cell during measurement.

Content

Diffuse Reflectance Measurements and Simulations for In-Line Characterizing Concentrated Liquid Dispersions

Sebastian Boldt[1]; Prof. Dr.-Ing. Hermann Nirschl[1]; Ouwen Zhai[1]; Dr. Marco Gleiß[1]; Prof. Dr. Dr. Dietmar Lerche[2]
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Abstract

The study of diffuse reflection in particle-laden media began in 1905 with theoretical investigations that provided critical insights into optical spectra and their application in planetary system analysis. These early contributions laid the groundwork for modern advancements in astrophysics and radiative transfer, influencing diverse fields such as medicine and technology. By 1914, the radiative transfer equation was formalized, and subsequent developments in the 1950s introduced refined solutions. Over time, diffuse reflection has become an essential tool in chemical analysis, particularly since the mid-20th century. Technological innovations, including Monte Carlo methods and studies of optical properties in turbid media, have enabled precise analysis of particle size, suspension concentration, surface textures, and thin films. Such progress is integral to autonomous systems, where real-time, non-contact measurements are vital for monitoring quality parameters.

This study investigates the theory of diffuse reflectance by combining simulations with experimental data and introduces an innovative measurement technique for in-situ monitoring of product quality parameters. The methodology is validated through diverse applications. To establish correlations between the parameters of interest—specifically dispersed mass concentration and particle size distributions—data-driven regression as well as compartment and physical models are utilized for inverse radiative photon transport calculations and optical property estimations.

Content

CDMA: Centrifugal Differential Mobility Analyzer – Measurement of two-dimensional particle property distributions

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Abstract

The CDMA ("Centrifugal Differential Mobility Analyser") was developed to determine a two-dimensional particle size distribution of a particle collective with a single, compact device. Classification occurs in the annular gap between two concentrically arranged cylinders – analogous to the DMA. The Particle classification is carried out by using an applied electric field and a centrifugal field applied simultaneously by rotation. The variation of applied voltage and rotational frequency enables the determination of the complete two-dimensional property distribution of aerodynamic and mobility equivalent diameter. Since the evaluation of the measurement signal results in a poorly conditioned system of equations, a back calculation employing the "Pojections Onto Convex Sets" (POCS) method is carried out. The presentation shows the prototype of the classifier (CDMA), the entire measurement setup and the methodology of the inversion calculation. The classifier is comprehensively characterised. Model calculations for inversion are presented, which demonstrate the robustness of the method even to superimposed stochastic errors. Initial measurement results from real particle collectives of aerosols from silver particles and halite in various agglomeration states are shown. In addition, the two-dimensional results obtained with the measuring apparatus are used to infer particle properties such as shape factors from the two-dimensional particle size distributions obtained.

Content

Pneumatic Conveying of Hydrated Lime: Between Anecdotal Experience and Rigorous Research Efforts – An Exploration of the State of the Art

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Abstract

Pneumatic Conveying of Hydrated Lime: A Problematic Substance Between Anecdotal Experience and Foundational Research Efforts – A Review of the State of the Art

Pneumatic conveying of hydrated lime (Ca(OH)2) is a critical component in flue gas treatment systems in power plants and waste incineration facilities. Despite well-established standards for conveying parameters, persistent challenges indicate that adherence to guidelines does not always guarantee optimal operation. This review aims to explore the intricate issues associated with the pneumatic transport of hydrated lime, with a particular focus on deposit formation and the triboelectric charging of particles. The research identifies two primary types of deposits: fluffy, easily removable agglomerates and hard, cement-like aggregates. By developing an ontology that models the factors affecting these formations—such as airflow velocity, humidity, and triboelectric effects—a comprehensive and extendable framework is provided for analysing these interactions. This review draws on extensive literature and technical data to propose strategies for optimizing conveying processes, such as adjusting air velocity and load, utilizing conductive materials, and refining system design to minimize disruptions. While our study enhances the understanding of pneumatic conveying of hydrated lime, it highlights the necessity for further quantitative research to precisely establish optimal parameters. The manuscript advocates for the use of computational simulations, including CFD (Computational Fluid Dynamics) and DEM (Discrete Element Method), to make more accurate predictions regarding conveying behaviours under diverse conditions. It accentuates contemporary challenges and proffers prospective solutions to enhance the efficiency and dependability of hydrated lime conveying systems, with the objective of forging stronger connections between experiential knowledge and scientific rigor.

Content

1. Introduction

Effective flue gas treatment is a cornerstone of environmental efforts to achieve cleaner air. At the heart of these processes, particularly in smaller combustion plants, is the use of dry sorption methods with hydrated lime (Ca(OH)2) for pollutant mitigation. This process is valued for its cost-effectiveness and relative simplicity, making it particularly suitable for smaller installations. Success in these systems hinges on the efficient and reliable delivery of powdered sorbent, predominantly through pneumatic dilute-phase conveying. This method is lauded for its simplicity and flexibility, aiming to achieve homogeneous sorbent dispersion for maximum reaction efficiency.
Despite decades of empirical guidelines defining parameters such as conveying velocities and solids loading, numerous plants labelled as "problematic" exhibit inconsistent operations. These discrepancies, observed even among identical facilities, suggest complexities that transcend traditional wisdom. Modern demands for environmental and economic efficiency necessitate a dynamic operation of flue gas treatment systems, adapting injection rates to variable pollutant loads. This necessity frequently pushes conveying systems beyond steady-state conditions, breaching recommended parameter limits. While such variability can be beneficial, it intensifies existing issues and introduces new challenges.

In-depth reviews identify substantial gaps in understanding, as conventional explanations focusing on insufficient loading or high velocities prove inadequate. A significant factor emerging from recent research is triboelectric charging—a process involving charge transfer between materials upon contact and separation. This often underestimated phenomenon can dramatically alter particle adhesion and cohesion, directly affecting conveying behaviour and deposit formation.
This review aims to bridge the gap between empirical observations and scientific knowledge by examining the interplay of practical experiences and foundational research, emphasizing the of triboelectric effects in hydrated lime transport. Such insights are critical for developing more robust and reliable system designs, ensuring both innovative and sustainable operation.

2. Problem Description

The pneumatic dilute-phase conveying of hydrated lime poses consistent challenges, leading to the formation of deposits within pipelines and system fittings. These deposits often

accumulate rapidly, resulting in operational disruptions that necessitate demanding mechanical removal efforts. Pioneering studies such as [She-11], as well as contributions from [Beh-99] and [Sch-12], have extensively documented these issues, attributing them to factors like low solids loading and high conveying velocities.

These deposits constrain the conveying path, leading to measurable increases in pressure. While spontaneous detachment of deposits occasionally provides temporary relief, many systems experience escalating pressure fluctuations. These spikes compromise dosing accuracy and worsen problems related to powder-laden leakage air within rotary valve systems. Left unaddressed, these issues can halt conveying flows entirely, leading to extensive clean-up efforts and downtime, with obstructions forming within just hours.

Two primary deposit types have been identified:

- Type 1: Fluffy, soft, and light agglomerates. These loosely bound particles are generally manageable through routine purging and the use of sweeping grit in combination with elastic hose lines that facilitate self-cleaning via vibration and deformation [Beh-99], [Sch-12].
- Type 2: Concrete-like, firm aggregates. These dense plaques are difficult to dislodge and often require significant mechanical force or line dismantling. While managing "Type 2" formation presents challenges, strategic design can encourage their localization to more accessible areas.

Notably, both deposit types may occur simultaneously at different locations within a single pipeline, highlighting the significance of local conditions. Chemical analyses indicate that fresh deposits often mirror the composition of the original hydrated lime [She-11], suggesting that initial deposition does not necessarily involve significant chemical changes. However, over time, deposition may lead to hardening into limestone via carbonation, eventually detectable through chemical means.

A comprehensive understanding of these deposit types is critical for developing effective mitigation strategies. It necessitates an exploration of the interplay of operational practices, environmental influences, and particle interactions, thus guiding the engineering of more robust pneumatic conveying systems.

3. Common Methods for Problem Resolution and Mitigation

The pneumatic dilute-phase conveying systems for hydrated lime are rarely free from issues, necessitating routine inspection and cleaning to maintain operation. The absence of a "Best Available Technology Reference" for perfectly stable conveying results in varied plant

designs and persistent optimization challenges, even among systems from established manufacturers.

Key Recommendations (Behrendt [Beh-99], Schneider [Sch-12], [Sch-13]):

- Solids Loading (μ): Maintain ratios of 1 to 2, with a maximum superficial air velocity of v=15 m/s.
- Elastic Conveying Lines: Promote self-cleaning through natural movement dynamics.
- Temperature and Humidity Control: Mitigate effects by increasing line pressure or using heating elements. Indoor silo housing and controlled temperature environments yield similar benefits.
- Material Aging: Limit hydrated lime storage to a maximum of 14 days; apply BET 20 grades instead of BET 40 in case of issues, and prioritize fresh deliveries.
- Pressure Regulation: Keep pressure differentials across the line below p=300 mbar.
- Simplified Conveying Path Design: Ensure routes are short and uncomplicated, with pulsation-free dosing.

These recommendations are most effective when implemented collectively, as addressing isolated factors is often insufficient given the complex interplay of multiple variables.

Effective Adjustments and Strategies:

- Air Speed and Loading Adjustments: Modify blower speed or conveying cross-sections to improve performance.
- Environmental Controls: Enclose silos, utilize heating, insulate setups, or source blower air from indoors to maintain optimal conditions.
- Material Handling Improvements: Use mechanical discharge aids or less reactive hydrated lime products.

Anecdotal and Advanced Solutions:

- Replacement of specific components (e.g., rotary valves, screw conveyors, blowers).
- Modifying conveying air temperature with heating/cooling or insulation.
- Applying Teflon coatings, controlling dew points, or using humidification to address static charges.
- Utilizing mechanical knockers or vibrators, adjusting admixture ratios, or changing suppliers.

While coherent methodological solutions are elusive beyond a singular focus on air velocity [She-11], strategies to decrease cohesion and enhance conductivity are recognized, such as lignosulfonate [Fan-95], alcohol [Pfe-08], or targeted air ionization.

Maintaining stable line pressure is crucial for operational safety; fluctuations require immediate investigation and documentation in a disturbance logbook to facilitate systematic analysis. Ultimately, pneumatic conveying design should be integrated into early plant planning to mitigate potential failure impacts effectively.

4. Conveying-Relevant Properties of Hydrated Lime

A comprehensive understanding of hydrated lime's properties is crucial for its effective pneumatic conveying. Hydrated lime ($Ca(OH)_2$), produced from limestone ($CaCO_3$) through calcination and slaking, exhibits variability in structure and properties due to the natural variation in limestone origins, different manufacturing processes (such as kiln types), and manufacturer-specific practices.

In flue gas treatment applications, the reactive surface area of hydrated lime is a critical property. While particle fineness is noteworthy, the primary determinants of the reactive surface area are particle porosity, roughness, and morphology. Highly porous or irregularly shaped particles enhance the reactive surface area. This is typically quantified using the BET method (DIN 66 131), which measures monomolecular gas adsorption, such as with nitrogen.

Hydrated lime particles generally have a median size (d_{50}) below 10 µm, placing them in Geldart Class C [Gel-73]. Such fine particles pose challenges in conveying due to dominant interparticle cohesive forces [Mol-82]. Van der Waals forces impact short-range particle interactions, while triboelectric charging can influence behaviour over longer ranges, such as centimetres, significantly affecting pneumatic transport [Fan-95]. These forces lead to phenomena such as cohesion, adhesion, and bridging, although dry materials may still flow adequately.

Material aging is a known issue; hydrated lime exposed to air reacts with CO_2 to form limestone ($CaCO_3$) and water:

$$Ca(OH)_2 + CO_2 \rightarrow CaCO_3 + H_2O$$

This reaction creates surface moisture, increasing adhesiveness and cohesion, reducing flowability, and facilitating caking. B. Chen et al. [Che-04] demonstrated that this carbonation primarily affects the particle surface, with water penetrating inward, thereby promoting reactions and precipitating $CaCO_3$ on the periphery. The result is a layered structure with a $Ca(OH)_2$ core and a fine $CaCO_3$ shell, explaining why fresh deposits resemble the chemical composition of the original material, while significant limestone formation is noted in aged, hardened deposits.

Triboelectric charging is a critical factor during the pneumatic conveying of both hydrated lime and limestone [She-11], [Fan-95]. Initially, it's assumed that hydrated lime particles gain negative charges and limestone particles positive. However, studies such as [Zha-03] highlight a more nuanced bipolar charging scenario in polydisperse powders, where larger particles tend to acquire positive charges and smaller particles negative ones, challenging simplistic models [Kam-23]. This charging behaviour is influenced by factors such as pipeline materials, loading, transport/air velocity, grounding, air humidity, surface structure, and impurities [Fan-95], [Lee-93]. A deep understanding of these interactions is crucial for effective mitigation strategies.

5. Influence Studies by L.-S. Fan and B. Sherer

The pioneering work of L.-S. Fan [Fan-95] revolutionized the understanding of hydrated lime conveying by highlighting triboelectric charging as a principal factor in adhesion and deposit formation. Fan's research established a foundational basis for considering electrostatic interactions, extending beyond traditional mechanical or moisture-related issues.
Building on Fan’s insights, B. Sherer's extensive laboratory study [She-11] systematically explored the effects of air velocity, solids loading, air humidity, and temperature on deposit behaviour. His experiments revealed distinct characteristics of the two primary deposit types:
- "Type 2" deposits (hard aggregates) were found to form more intensely in the inlet region of the experimental injector's catch nozzle. This region experienced sharp increases in local air velocity and redirection of the particle stream, resulting in heightened particle-wall contact and triboelectric charging—effectively supporting Fan’s hypothesis. The combination of intense mechanical forces and charge accumulation in these areas facilitates the formation of robust deposits.
- "Type 1" deposits (fluffy agglomerates) appeared to be independent of direct carbon dioxide influence, suggesting that their primary mechanism is not chemical carbonation. Instead, these deposits likely stem from inter-particle cohesion affected by trace moisture or inherent particle stickiness, exacerbated by electrostatic forces but lacking significant hardening.
Both studies emphasized the crucial of conveying line material selection, as different materials exhibit varying degrees of electron affinity, thus influencing charge transfer. Steel, with its neutral tendency, contrasts with materials like copper and brass, which tend to accumulate stronger charges, thereby enhancing electrostatic adhesion. Pure plastic materials, notably those without conductive formulations, are particularly susceptible to

charge build-up due to their insulating properties. This underscores the importance of choosing materials that minimize charge generation or enable effective dissipation.
Together, Fan and Sherer's research provides compelling evidence that triboelectric charging is a primary driver of deposit formation, influenced significantly by operational parameters affecting particle-wall interactions. Their work integrates physical, chemical, and electrical phenomena, offering a deeper understanding of the complex mechanisms behind deposit formation in pneumatic conveying systems

6. Approaches to Phenomenological Model Building

The traditional "chemical" explanatory model, predominantly advocated by K. Schneider and J. Behrendt in their works such as [Beh-99] and [Sch-12], emerged from a combination of unpublished conveying trials conducted at Steinmüller/Gummersbach in the late 1980s, extensive experience gained from numerous installed and optimized plants, and their own insightful anecdotal observations. This model posited that the primary cause of conveying problems, particularly deposit formation, was rooted in a carbonation reaction (Effect 4), specifically the conversion of hydrated lime to limestone. The model further suggested that this carbonation was either initiated or significantly promoted by an "initial energy" imparted through impacts of particles with the pipe wall. According to this theory, the propensity for deposit formation was primarily dependent on two factors: the age of the sorbent (as older material would have undergone more pre-carbonation) and the frequency and intensity of particle-wall collisions. The idea was that these collisions provided the necessary mechanical energy to activate or accelerate the chemical conversion at the particle surface, leading to hardened deposits.
However, a more detailed and holistic analysis, incorporating the crucial insights from Chapters 4 and 5 regarding material properties, aging, and especially triboelectric charging, leads to the conclusion that this purely "chemical explanation" with its concept of mechanically induced "initial energy" is, in isolation, insufficient and not fully sustainable. While carbonation certainly plays a role in the hardening of deposits over time, it does not adequately explain the initial formation or the specific characteristics of all deposit types. The model, therefore, requires significant supplementation to fully account for the observed phenomena. The complex interplay of various factors dictates the ultimate behaviour of the system, making a singular explanation inadequate.
In an effort to construct a more comprehensive phenomenological model, we attempt to systematically categorize and correlate the observed phenomena with their underlying

influences and effects. This structured approach allows for a clearer understanding of the complex interactions at play, moving beyond a simplistic cause-and-effect relationship. This framework serves as an extensible ontology, allowing future research to integrate new findings and refine the relationships described.

The Phenomena to be explained by the model are distinct manifestations of conveying problems:

Phenomenon (A) The fallout of particles from the conveying stream: This refers to particles losing their ability to remain suspended in the air flow, eventually settling at the bottom of horizontal sections or accumulating at bends. This can be due to a loss of kinetic energy or the formation of agglomerates that become too heavy to be transported.

Phenomenon (B) The formation of fluffy, removable agglomerates: These are the "Type 1" deposits, characterized by their loose, friable structure. They are relatively easy to dislodge, often forming soft clusters that can build up but are less tenacious than "Type 2" deposits.

Phenomenon (C) The formation of concrete-like aggregates: These are the "Type 2" deposits, distinguished by their extreme hardness and tenacious adherence to pipe walls. Their removal often requires significant mechanical force and can lead to extensive downtime.

Phenomenon (D) The adhesion of these agglomerates and aggregates to the pipe wall: This specifically addresses the mechanism by which the formed clusters (B) or hardened masses (C) attach and accumulate on the internal surfaces of the conveying line.

These phenomena are subject to the following Influences – the variables and conditions that drive or modify the observed behaviours:

Influence (a) Loading of the conveying air (Solids Loading Ratio): This is the mass ratio of hydrated lime to conveying air. A higher loading means a greater concentration of particles in the air stream, impacting particle-particle and particle-wall collision frequencies.

Influence (b) Air velocity of the conveying air (Superficial Air Velocity): The speed at which the air moves through the pipe. This directly affects the kinetic energy of the particles, their collision intensity, and the overall aerodynamic forces acting on them.

Influence (c) Air humidity of the conveying air: The moisture content in the conveying air. This is a critical influence, as it affects surface conductivity, capillary forces, and chemical reaction rates.

Influence (d) Temperature: The ambient and conveying air temperature. This impacts relative humidity, material reactivity, and the physical properties of the particles themselves.

Influence (e) Conveying line geometry: The physical design of the pipeline, including bends, elbows, expansions, contractions, and overall length. These features influence flow patterns, turbulence, and particle trajectories.
Influence (f) Conveying line material: The intrinsic material of the pipe (e.g., steel, various plastics, copper, brass). Different materials have distinct surface properties, coefficients of friction, and positions in the triboelectric series.
Influence (g) Reactive surface area (BET) of the sorbent: The specific surface area of the hydrated lime particles, measured by the BET method. This property directly correlates with the material's chemical reactivity and available surface for intermolecular forces.
Influence (h) CO2 content of the conveying air: The concentration of carbon dioxide in the air used for transport. This directly influences the rate of the carbonation reaction, especially in the presence of moisture.
These influences, in turn, lead to or modify the following Effects – the underlying physical and chemical mechanisms that cause the observed phenomena:
Effect (1) Electrostatic forces in the cm-distance range: These are long-range attractive or repulsive forces arising from triboelectric charging. They are significant even when particles are not in direct contact and can cause particles to move towards or away from pipe walls.
Effect (2) Van der Waals forces in contact: These are short-range attractive forces that become dominant when particles are in very close proximity or direct physical contact. They contribute to particle cohesion and adhesion.
Effect (3) Capillary action in contact: Attractive forces due to the formation of liquid bridges between particles or between particles and the wall, caused by condensed moisture. These forces can significantly increase adhesion and cohesion.
Effect (4) Chemical bonding in contact / Carbonation: The formation of strong chemical bonds, particularly through the conversion of hydrated lime to calcium carbonate (limestone). This leads to the hardening and cementing of deposits over time.
Effect (5) Flow forces: Aerodynamic drag and lift forces exerted by the conveying air on the particles. These forces are essential for particle suspension and transport.
Effect (6) Gravity: The omnipresent downward force acting on particles, which, if not overcome by flow forces, leads to settling or stratification within the pipe.
By mapping these elements, a more nuanced understanding emerges. Let's consider some key interactions:
Phenomenon A (Particle Fallout): This is primarily influenced by a lack of sufficient Flow forces (5) to counteract Gravity (6). However, the preceding formation of Agglomerates "Type 1" (B) (driven by Effects 2 and 3) can create oversized particles that become too heavy for

the air stream, leading to their fallout. Furthermore, if the air velocity (Influence b) falls below the saltation velocity or minimum transport velocity, particles will inevitably drop out. Charge accumulation on particles and walls (Effect 1) can also exacerbate fallout if particles aggregate into larger, heavier clusters or if electrostatic forces cause particles to adhere to the bottom of the pipe in stagnant zones.

Phenomenon B (Fluffy Agglomerates): The formation of these loose agglomerates is strongly dependent on Capillary action (3), especially if even trace amounts of moisture are present (Influence c). Additionally, Van der Waals forces (2) upon particle-particle contact contribute significantly. Mechanical agitation, such as that imparted by screw conveyors or rotary valves during dosing, can intensify these contacts, promoting initial agglomeration.

Phenomenon C (Concrete-like Aggregates): This is the more complex and problematic deposit type. Here, Chemical bonding/Carbonation (4) plays a significant role, driven by influences such as humidity (c), temperature (d), CO_2 content (h), and the material's reactive surface area (g). However, Electrostatic forces (1) (influenced by a, b, c, f) are crucial for the initial collection and compaction of particles that then undergo subsequent carbonation. The high kinetic energy from high air velocity (Influence b) can lead to more impactful collisions, increasing charge transfer and initiating strong electrostatic attraction, which facilitates the formation of these hard, tenacious aggregates.

Phenomenon D (Adhesion to Wall): Both Electrostatic forces (1) (Influences a, b, c, f) and Van der Waals forces (2) are key mechanisms for particles sticking to the pipe wall. When moisture is present (Influence c), Capillary action (3) can significantly enhance this adhesion. Once an initial layer is formed, Chemical bonding (4) (i.e., carbonation) can then solidify these layers into extremely hard and difficult-to-remove deposits.

A particular complexity arises with triboelectric charging of polydisperse powders. Scientific literature, such as [Zha-03], has reported that such powders can exhibit bipolar charging, where different size fractions acquire opposing charges. For example, [Zha-03] observed that larger particles often acquire positive charges, while smaller particles tend to acquire negative charges in fluidized beds. This observation directly challenges the simplified assignment by [Fan-95] that limestone ($CaCO_3$) particles are positive and hydrated lime ($Ca(OH)_2$) particles are negative. The reality is significantly more complex, as illustrated by more recent research like [Kam-23], which suggests that particle morphology, surface chemistry, and even minor impurities can profoundly influence charge acquisition. This complexity means that the interactions within the conveying line are not just between uniformly charged particles but a dynamic interplay of positive, negative, and potentially

neutral particle fractions, leading to complex aggregation behaviours and a greater propensity for adhesion to oppositely charged wall sections or already deposited layers.

Interactions between Influences and Effects (Illustrative Examples):

Let's delve into some specific, often counter-intuitive, interactions between influences and effects:

Air Humidity (c): This influence has a multifaceted impact. On one hand, higher air humidity increases the electrical conductivity of the air-product mixture, which in turn facilitates the dissipation of accumulated charges. This leads to a reduction in triboelectric charging (Effect 1), thereby mitigating electrostatic adhesion. On the other hand, increased humidity significantly enhances capillary action (Effect 3) between particles and between particles and the wall. This leads to increased cohesion between particles, making the powder "stickier" and worsening its flowability. Furthermore, humidity acts as a crucial reactant and medium for the carbonation reaction (Effect 4), accelerating the chemical conversion of hydrated lime to limestone, which directly contributes to the hardening of deposits. Thus, humidity presents a delicate balance: it can reduce one major problem (electrostatic adhesion) while exacerbating others (cohesion and chemical hardening). The "optimal" humidity range is therefore very narrow and depends heavily on other operating conditions. For example, [She-11] noted that a moderate increase in humidity can reduce deposition by mitigating static charge, but excessive humidity leads to film formation and poorer flow.

Temperature (d): Temperature also exerts complex, interwoven effects. A higher temperature generally reduces the relative humidity of the conveying air for a given absolute moisture content. This reduction in relative humidity lowers the electrical conductivity of the air, which can in turn increase the severity of triboelectric charging (Effect 1). Paradoxically, higher temperatures can also reduce the surface tension of any condensed water film, thereby decreasing capillary action (Effect 3). Regarding deposit types, Sherer's findings suggest a correlation where low temperatures favor "Type 1" (fluffy) deposits, likely due to increased relative humidity and capillary forces, while high temperatures tend to promote "Type 2" (hard) deposits, potentially due to accelerated carbonation (Effect 4) and possibly intensified electrostatic effects in drier conditions.

Loading of the conveying air (a): Increasing the solids loading ratio has multiple beneficial effects. A higher loading leads to a greater spatial concentration of particles, resulting in more frequent particle-particle collisions. These collisions provide more opportunities for charge equalization (Effect 1) between particles, effectively lowering the overall triboelectric charge accumulation within the system. Simultaneously, a higher loading reduces the relative frequency of particle-wall contacts for a given mass flow, thus minimizing the primary source

of charge generation. This also reduces the influence of van der Waals forces (Effect 2) and capillary action (Effect 3) by reducing the contact time with the wall surface and promoting particle-particle interactions.
The development of such a phenomenological model, while not yet fully quantitative, provides a crucial framework for understanding the intertwined physical and chemical processes. It moves beyond isolated explanations and emphasizes the synergistic effects that lead to operational problems in hydrated lime conveying. This understanding is a prerequisite for developing more effective and predictive design and operational guidelines. The utility of this model lies in its ability to systematically map known influences to their complex effects, guiding future research toward quantitative relationships.

7. Summary

A purely fluid dynamic perspective is inadequate to fully understand the pneumatic conveying of fine powders such as hydrated lime. Our analysis confirms that triboelectric phenomena significantly impact the behaviour of these fine powders. It is evident that triboelectric charging has substantial effects on particle adhesion, agglomeration, and deposit formation within hydrated lime systems.
Simulations, such as those illustrated in [Gro-16], demonstrate a clear correlation between conveying air velocity and particle-wall contact frequency, which directly increases triboelectric charging. This finding corresponds with Muschelknautz's "turbulence power" concept, where elevated velocities and turbulence intensify charge generation.
The recommendations from J. Behrendt and K. Schneider - to maintain lower conveying velocities and increase the solids loading ratio - are empirically validated. Their effectiveness arises primarily from mitigating triboelectric charging rather than merely reducing chemical reactions. Higher loading decreases the relative frequency of particle-wall contacts compared to particle-particle contacts, aiding in charge dissipation, while lower velocities lessen impact frequency and charge generation. Charge build-up directly enhances the affinity of particles for surfaces, leading to adhesion and deposits.
We hypothesise that at increased air velocities, additional charge accumulation results in differentiated electrostatic charging of particles, possibly due to varying $CaCO_3$ surface formations resulting from ageing. These oppositely charged particles subsequently attract each other, leading to sintering and the formation of stubborn "Type-2" deposits. The combined effects of chemical hardening and strong electrostatic attraction account for their extreme durability.

Despite our improved understanding, current models lack the quantitative precision necessary for optimised process parameters. This highlights a pressing need for further research. Advanced numerical simulation methods, such as Computational Fluid Dynamics (CFD) for airflow, the Discrete Element Method (DEM) for particle tracking, and Direct Numerical Simulation (DNS) for microscopic interactions, represent promising approaches for accurate behavioural predictions under varied conditions.

Even with these challenges, the current knowledge facilitates targeted improvements in conveying quality. Beyond traditional fluid dynamic optimisation, electrostatics must be considered, not solely for ATEX safety, but also for conveying performance. This involves implementing charge management and dissipation strategies. We advocate a dissipative design for the entire conveying system, applicable even in non-ATEX areas, particularly for fine Geldart Group C powders. This includes using conductive materials, ensuring proper grounding, and potentially employing active charge neutralisation techniques.

Triboelectric charging is inherent to frequent particle-surface contacts in components like screw conveyors and rotary valves. To mitigate this, charge equalisation measures are crucial. These might include the incorporation of conductive elements in dosing equipment, system designs that minimise high-impact collisions, or the controlled humidification of conveying air to improve conductivity. By integrating these deeper electrostatic insights, we can significantly enhance the efficiency and reliability of hydrated lime conveying systems, thereby strengthening the link between practical experience and scientific rigour.

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Pressure loss prediction of powder pneumatic conveying systems in straight pipes and bends and development of new pressure model utilizing Euler-Euler approach

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Abstract

This study illustrates the ability of computational fluid dynamics (CFD) to predict and analyze gas-solid flow behavior in pneumatic conveying systems (PCS) for fine powder transport, specifically focusing on fluidized dense-phase pneumatic conveying of fly ash (mean particle size: 30 μm, true density: 2,096 kg/m³). Employing the Euler-Euler approach, which models both phases as interpenetrating continua, we simulated flow transitions in horizontal pipes and predicted pressure losses in 90-degree bends using two commercial CFD platforms: COMSOL Multiphysics and Ansys Fluent.

To this end, CFD simulations were conducted to predict two-phase flow interactions and investigate flow behavior in PCS using the Euler-Euler approach, which aligns closely with the studies of Gidaspow. Sensitivity analyses were performed using the Kinetic Theory of Granular Flow (KTGF) and the Mixture Model, examining various velocity profiles, solid volume fractions, and mean particle diameters. The present study was validated against experimental research by Alkassar et al., demonstrating strong agreement with the simulated case studies.

To assess pressure losses in bends, multiple case studies explored how variations in flow velocities, volume fractions, and particle size distributions influence pressure losses. The study also examined bend orientations in horizontal and vertical configurations, determining initial acceleration and re-acceleration lengths through simulations using distinct equation-based approaches in COMSOL and Ansys Fluent. Results indicate that Ansys Fluent, leveraging the KTGF model, provides more accurate predictions of two-phase stream regeneration toward a developed flow field, closely matching the experimental findings of Pan and Wypych, which reported a dense flow regime length of 3–4 meters.

Content

Moreover, a novel non-dimensional pressure drop model was developed, incorporating solids loading ratio, volume fraction, and mean particle diameter—three key factors in industrial conveying systems. This model predicts pressure drop along horizontal pipes with enhanced accuracy compared to existing models, offering deeper insights into bulk solid flow behavior and flow formation patterns at each pipe section following bends. The model improves the design of industrial PCS by providing a practical tool for pressure loss estimation, facilitating time- and cost-efficient management through robust, reliable computer simulations, as opposed to experimental or operational approaches in test benches or plants.

Eccentric discharge behaviour in the small-scale model silo of a silo centrifuge

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Abstract

Although the development of silo construction began in the 19th century with the work of Janssen and following extensive research in the last century, many questions regarding flow in silos remain unanswered. Oversizing or, in the case of incorrect design, silo damage must be avoided, i.e. preventing dead zones is thereby one of the key factors. Since dead zones often cannot be fully avoided from a process engineering point of view, for example due to eccentric discharge of the bulk solids, it is even more important to understand their development, their effect on the bulk solids properties and the resulting loads on the silo wall. Generally, eccentric openings lead to asymmetrical load effects, thereby requiring special attention. In the current load approach for the design of eccentric discharge (DIN EN 1991-4), several assumptions are necessary. As a means of contributing to a sturdy and sustainable silo design without oversizing or silo damage, fundamental research is important. In this study, the influence of the position of the outlet opening with regard to the flow profile, the loads on the silo wall and the outlet mass flow is analysed using a silo centrifuge. The silo centrifuge used was developed in-house at the TU Braunschweig and enables the simulation of consolidation conditions of a large-scale silo in an accordingly small-scale model silo through centrifugal acceleration. Besides the outlet's eccentricity, the following factors from the silo centrifuge operation should be considered: the experimental setup (e.g., silo filling) and the outlet's position relative to the direction of rotation. The presentation introduces findings on the application of the Braunschweig silo centrifuge for researching the flow behaviour when using eccentric outlet openings.

Content

The evolution of continuous melt granulation: from one to two stages to decode the complexity?

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Abstract

Granulation is a central unit operation of powder processing to improve the handling properties of a bulk materials. In recent years, research focus shifted to melt granulation as hereby a subsequent drying step is not required and moisture-sensitive materials can be handled. Simultaneously, the focus of process technology focus shifted to continuous manufacturing to minimize quality fluctuations by an enhanced process control. Lately, the application of a planetary roller granulator has been introduced [1] as a promising alternative for continuous melt granulation with respect to the unique process concept. Systematic investigation revealed hereby, that a linking of the granulation performance to the process parameters was not feasible [2,3] due to a local and timely coupling of binder melting and model compound granulation during processing for a standard set-up.
Therefore, the aim of this work is to investigate a sophisticated two-stage continuous planetary roller melt granulation, whereby the two occurring unit-operations are locally and timely separated even though the overall process is executed in one machine.
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Challenges of Dry Coating: How do Fibrils and Powder Properties shape Electrode Properties for Lithium-Ion Batteries?

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Abstract

A key focus of current battery research is the production of lithium-ion batteries (LIBs) with high energy or power density. The state-of-the-art production process for their electrodes is a solvent-based film coating, which involves an energy-intensive drying and a solvent recovery step. An innovative alternative is the dry coating of LIBs, which avoids the use of solvents entirely. The dry mixing and by that also structuring of the powdered electrode components (active material, conductive additive, binder) represents the first step in this solvent-free electrode production process. In the second, film forming step using a horizontally orientated, multi-roll calendering device, the shear forces in the calender gap cause further fibrillation of the binder and thus the formation of a free-standing film.
In general, dry coating presents new challenges in terms of process-structure-property relationships for graphite anode and NCM cathodes regarding the binder PTFE and the resulting electrode properties. Various characterization methods were used to investigate the influence of batch mixing (Zeppelin FML10) and the film formation parameters using a multi-step calendering on the resulting PTFE fibrillation within anode and cathode powders and the resulting free-standing films (e.g. particle size distribution, compaction speed, porosity and electrical resistance). Initial results showed that the temperature during binder addition, the mixing time at constant shear forces, and the post-treatment significantly influence the PTFE fibril structure and powder properties, such as granule size distribution and flowability. The discussion explores, how resulting fibril network and powder properties influence the processability of free-standing films and electrodes.

Content

Particle Processing in Solid-State Battery Cathode Development: A Physico-Chemical Perspective

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Abstract

Solid-state batteries (SSBs) are being extensively studied for their potential to deliver higher energy and power density with improved safety compared to liquid-electrolyte lithium-ion batteries. Laboratory-scale research often relies on studies conducted with pelletized press cells; however, a recent interlaboratory round-robin study revealed substantial discrepancies in cell assembly and performance among research groups
Our work highlights the importance of the dry particle mixing procedure for cathode composite preparation, which is often conducted via hand mortaring at the lab scale. This process accounts for many of the variations observed in the achievable capacities of cells fabricated with identical materials and assembly parameters. By employing a mini vibrating mill for machine-based mixing, we demonstrate a significant improvement in reproducibility. To evaluate the mixing quality, we introduce the in-situ electrochemical open-circuit relaxation method, which quantifies the cathode active material (CAM) utilization as a valuable figure of merit. These results emphasize the critical importance of the particle mixing process to ensure reliable electrochemical analyses.
We further investigated the production of cathode composites in a scalable high-intensity mixer, applying the same laboratory-scale principles to evaluate the electrochemical performance. A mixed-conducting matrix coating is presented as a process design approach for cathode composites, illustrating the role of dry particle coatings for SSB cathodes. Overall, our findings underscore the challenges and opportunities in particle mixing and coating for SSB cathodes, stressing the need for further research in this field of particle technology.

Content

Engineering electronic pathways within cathode micro-structures through carbon-coated NMC via dry mixing

Dr. Guo Jung Lian PhD; Jak Hudson; Dr. Denis Cumming PhD; Prof. Rachel Smith PhD
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Abstract

Cathode performance in lithium-ion batteries is often limited by the low electronic conductivity of active materials. To address this, conductive additives (e.g., carbon black) are commonly used to enhance the electronic conductivity within electrode structures. However, their inactive nature reduces energy density and is therefore kept to a minimum, typically within 2 wt% in commercial electrodes. The conventional wet slurry mixing process often results in an uncontrolled distribution of active material, conductive additives and binder, making it challenging to control the quantity and distribution of conductive additives within the cathode. Studies have suggested the need for strategic distribution of conductive additives to form both long and short-range electronic pathways for improved conductivity. Recent literature has explored the use of a novel dry mixing technique, mechanofusion, as a pre-processing technique to apply coatings to active materials using high shear and compression forces. In this study, a novel approach to electrode design was demonstrated, by controlling the formation of short and long-range electronic pathways within cathodes via particle engineering. The relative combinations of the electronic pathways were controlled by assigning different fractions of the total conductive additive content in dry mixing and conventional wet mixing. The resulting slurry, electrode and cell performance were studied to assess the impact of these different electronic pathways on electronic conductivity and electrochemical behaviour. By developing a comprehensive understanding of the interplay between material properties, manufacturing processes and final cell performance, this work provides valuable insights into design strategies for next-generation electrodes.

Content

On the Role of Polymer Binders for the Electrochemical Properties of Dry-Processed Electrodes

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Abstract

Lithium-ion batteries is assuming an increasingly critical function in the transition to sustainable energy solutions. Traditional wet manufacturing processes for battery electrodes pose environmental challenges due to toxic solvent used and requires 100-meter-long drying facilities. In response, dry manufacturing processes are gaining attention for their potential to reduce ecological impact comparing with traditional process. In contract with matured wet process technologies in battery industry, the new process relies highly on integration of various particle technologies, including powder homogeneous mixing, milling, grinding etc. In addition to the novelty particle process, the commonly used binder, PTFE, in the process isn't ideal due to its low mechanical and electrochemical stability. To overcome the binder issue, this talk seeks alternative additives and investigate the molecular behavior of the additive particles along the processing stages and uncover the critical particle parameters to improve the dry electrode process.

Content

Engineering Cellulose Nanofiber-Fe3O4 Nanocomposites for Superior Magnetic Responsiveness and Binding Affinity in Biomedical Innovations

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Hiroshima University

Abstract

1. Introduction
TEMPO-oxidized cellulose nanofibers (TOCN), produced through controlled oxidation of cellulose using TEMPO, are gaining prominence in the biomedical field due to their high carboxyl content (1.8 mmol/g) and strong negative surface charge (-40 mV).1 These properties make TOCN highly effective in adsorbing positively charged substances.2,3 However, separating TOCN from liquid media is challenging as centrifugation causes particle aggregation, reducing reusability.
To address this, TOCN was combined with 15 nm Fe3O4 nanoparticles (NPs) to create TOCN- Fe3O4 (TF) composite particles, enabling efficient magnetic separation. This study utilized a one-step spray drying method to synthesize TF particles while varying Fe3O4 concentrations.4 The composites' morphology, magnetic properties, separation efficiency, and protein binding performance were evaluated. The carboxylate groups on TOCN allowed covalent antibody attachment, with biotin served as the antibody (protein) and streptavidin as the antigen.

2. Methods
TF composite particles were synthesized via spray drying using a two-fluid nozzle. The precursor solution contained 2 wt% TOCN, an oxazoline-based cross-linker 5, and 15 nm Fe3O4 NPs at mass ratios of 0.1 to 3 relative to TOCN. Spray drying was performed at 120 °C with a liquid flow rate of 2.5 mL/min, spray gas flow rate of 6.0 L/min, and carrier gas flow rate of 583 L/min. The particles were characterized for morphology, zeta potential, and magnetic properties. Protein binding performance was evaluated using a streptavidin-biotin system to assess biomedical potential.

3. Results
The morphology of TF composite particles, synthesized by spray drying with varying mass ratios of 15 nm Fe3O4 NPs. Increasing Fe3O4 concentration transformed particle morphology from wrinkled to spherical due to NP aggregation on the surface, preventing TOCN entanglement. The TF-1 sample in deionized water, initially well-dispersed and stable. Upon magnetic application, particles were attracted and accumulated, demonstrating strong responsiveness to an external magnetic field and confirming effective magnetic separation.

4. Conclusion
This study synthesized TOCN-Fe3O4 composite particles via spray drying, revealing concentration-dependent changes in morphology, surface charge, and magnetization. Higher Fe3O4 content improved magnetic collection efficiency and responsiveness. The composites demonstrated effective protein binding, showcasing their biomedical potential.

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An AFM-based approach for quantification of guest particle deformation during mechano-fusion

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Abstract

During the mechano-fusion process for dry particle coating, (hetero)-aggregates are formed consisting of host particles coated by smaller guest particles. During this process, the latter are exposed to intense particle–particle interactions and particle–wall impacts, which lead to deformation of their original shape. These deformations on the nano- and microscale can heavily influence the effective macroscopic properties of the resulting coated particles. We present a method for quantification of shape deformation based on measurements acquired by atomic force microscopy and apply it to guest particles during mechano-fusion [1]. The 3D shape of guest particles is reconstructed by means of an ellipsoidal fit. Using these reconstructed shapes, we quantify the degree of deformation by comparing the aspect ratios of the ellipsoidal fits before and after mechano-fusion. Such a quantification enhances the understanding of how process-related parameters influence geometric descriptors of involved particles, which in turn impacts overall macroscopic properties of the material.

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On the importance of mixer stressing conditions during the production of solid-state battery cathodes

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Abstract

The development of efficient composite cathodes is a key challenge in the advancement of solid-state batteries. In the presented studies, the influence of mixer type, applied materials, and mixing conditions on the microstructural and electrochemical properties of solid-state battery cathode composites is investigated. Three material systems – LiNi0.83Co0.11Mn0.06O2 (NCM) – Li3InCl6 (LIC) – carbon black (CB), NCM – Li6PS5Cl (LPSCl), and LiFePO4 (LFP) – LIC – CB, were examined using two different mixing devices: a high-intensity mixer and a planetary ball mill.
The results demonstrate that the choice of material system, mixer type, and applied process parameters significantly affect particle size, crystallinity and the distribution of the solid electrolyte within the composite. For the NCM – LIC and LFP – LIC – CB material systems, in the high-intensity mixer a reduction in solid electrolyte particle size was observed with increasing mixing time and rotational speed, whereas a coating of was achieved for the NCM – LIC system. A decrease in electrolyte particle size and composite aggregate formation was achieved in the planetary ball mill, which led to enhancements in the electrochemical performance due to a better ionic network. However, excessive stress caused by high stress intensities resulted in a reduced performance due to a decrease in the crystallinity of the solid electrolyte.
Discrete Element Method simulations of the mixing processes provided additional insights into the stressing mechanisms acting on the materials and the processes occurring within the mill. Applying characteristic parameters derived from the DEM-simulations the solid-state cathode mixing process can be optimal designed based on only few experiments.

Content

Experimental investigations into a jet-based direct mixing process for solid state battery cathode production in a turbulent pipe flow

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Abstract

The efficient mixing of different materials at the primary particle level (hetero-agglomeration) can lead to close interactions between the components (hetero-contacts), which give the entire composite material new specific properties. One example of this is the battery research in the field of solid-state batteries (SSB), where such interactions can contribute to improving the electronic and ionic conduction paths within the cathode material [1,2].
This experimental study investigates a jet-based direct mixing process for the targeted agglomeration and structuring of submicron materials in the gas phase, with the aim of improving the homogeneity and functionality of the resulting composites. The current application focus is on the development of cathode materials for SSB in the submicron range. Lithium iron phosphate (LiFePO4, LFP) is used as the active material, lithium halide (Li3InCl6, LIC) as the solid electrolyte and carbon black (CB) as conductive additive. With two dispersing and dosing units (rotating brush disperser RBG 1000 ISD from Palas), the starting materials are broken down to the primary particle level and fed into a turbulent mixing zone as particle-laden aerosol jets. In the mixing zone, the different particle jets come into contact with each other, resulting in hetero-contacts and the formation of heterogeneous agglomerate structures (hetero-agglomerates). The particle collective then flows through a sequence of pipe segments in which turbulent flow causes further mixing and agglomerate formation.
Based on experimental and simulative preliminary work, the process was further developed in a targeted manner, taking into account key factors influencing hetero-agglomeration [3,4]. An extended pipe section increases the residence time and promotes agglomerate formation, while reduced pipe diameters lead to increased collision rates and higher mixing efficiency

Content

due to increased particle concentration and turbulence. An optimized mixing zone geometry also ensures defined entry angles for the jets and improves initial mixing.
A systematic methodology was developed to comprehensively characterize the mixing process. First, the dispersing and dosing units are analyzed by means of laser light diffraction (LLD) in order to identify influencing factors on the dispersion quality and the particle size distribution (PSD) of submicron cathode material. The optimum dispersion settings determined in this way serve as the basis for subsequent investigations of the overall process. During the mixing process, samples can be taken at several defined positions along the flow path. Two methods are used: Firstly, samples are deposited on scanning electron microscopy (SEM) stubs to analyze morphology and composition using SEM in combination with energy dispersive x-ray spectroscopy (EDX). Secondly, a partial stream is extracted with a cascade impactor (DLPI+ from Dekati) in order to determine a mass-related PSD of the particle collectives after a gravimetric evaluation. In parallel, an in-situ characterization using LLD is carried out at the same positions to measure the PSD of the aerosol jet in real time. The comparison of these measurement methods allows conclusions to be drawn about the formation of the agglomerates in the gas phase. The entire produced composite material can be deposited on filters and then comprehensively characterized. In addition to the analysis of the microstructure using SEM, the elemental composition is quantitatively determined using inductively coupled plasma optical emission spectrometry (ICP-OES) in order to draw conclusions about homogeneity and the degree of mixing. Furthermore, the electrochemical properties of the composites, in particular the specific capacity, are investigated to assess their suitability and functionality as battery material.
In an ongoing parameter study, key factors influencing the jet-based mixing process are being systematically investigated. Among other things, different geometries of the mixing zone are used to specifically analyze the influence of the entry angle on the initial mixing. In parallel, operational process parameters such as volume and mass flow are varied to evaluate their effect on mixing and agglomeration. In addition, different material combinations will be investigated as part of a pre-mixing process and a sequential composite production process. Furthermore, a third disperser will be integrated into the system (process with three aerosol jets) in order to specifically investigate its influence on the structuring of the composites. The aim is to compare the resulting mixtures with those from the two-jet process - particularly with regard to potentially more efficient initial mixing and improved hetero-agglomeration.

The results of the investigation of the dispersers using LLD showed that an increase in the shear stress is primarily achieved by a higher air volume flow, while the solids mass flow has only a minor influence on the dispersing effect and can be flexibly adapted to subsequent processes with a high mass concentration (e.g. [3]). Drying the starting material improves the dispersion quality as it prevents moisture-induced agglomeration. A stainless steel brush proved to be particularly effective in breaking up agglomerates and resulted in a finer PSD than a nylon brush. Short outlet tubes reduce re-agglomeration due to reduced residence times and minimize material losses as a result of lower particle deposits. At the same time, smaller pipe diameters promote the deposition of larger particles due to increased turbulence, which also contributes to a finer PSD.
Initial results of the SEM stub samples of the produced composites show that the increased particle concentration and flow turbulence as a result of the modified pipe dimensions lead to a significantly increased number of particle interactions. This favors the formation of hetero-agglomerates. An increase in the size of the agglomerates can also be observed along the pipe flow, which indicates increased contact formation and continuous agglomerate growth within the flow path.

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Formation and characterization of nanostructured TiO2-ZrO2 hetero-aggregates in opposed jet fluidized bed

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Abstract

Nanoparticles of different materials can be mixed via opposed jet fluidized beds by overcoming the cohesive forces between their primary particles, leading to the formation of hetero-aggregates. The gas accelerates these aggregates, causing collisions that break them into smaller units, ideally primary particles. These particles subsequently re-agglomerate into new structures, forming hetero-aggregates that incorporate freshly formed homo-aggregates of the other material. This continuous sequence of mixing, breaking, and re-aggregating generates nanostructured hetero-aggregates with high mixing quality, specific structures, and numerous hetero-contacts. The feasibility of opposed jet fluidized beds for producing TiO2-ZrO2 hetero-aggregates was demonstrated by analyzing process parameters and their influence on mixing quality. Feed composition ratios (1:1, 2:1, and 1:2), fluidization air pressures (0.5, 2.5, and 5.0 bar), and processing times (1 and 5 minutes) were evaluated. Mixing quality was assessed through SEM/EDX to determine the Ti atomic fraction, along with measurements of hetero-aggregate features such as circularity, roundness, fractal dimension, and porosity. The results indicated that mixing quality is primarily dependent on feed composition, with process time and pressure showing no significant influence. Shape analysis revealed circularity and roundness values of approximately 0.4 and 0.5, respectively, while 2D and 3D fractal dimensions averaged 1.76 and 2.25. The aggregates exhibited a high porosity of approximately 0.894, consistent with star-like shapes and structural gaps. These findings highlight the potential of opposed jet fluidized beds for creating highly porous and nanostructured hetero-aggregates.

Content

Assessment of the mixing quality of carbon black – silica hetero-aggregates

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Abstract

The urgent need for a reduction in the use of fossil fuels has led to an increasing importance of electrical storage in the form of lithium ion batteries. Carbon black is added therein to improve the conductivity by ensuring electrical pathways through binder polymers and active material. Therefore, high dispersion on nanoscale levels is necessary. However, during dry mixing cluster formation and aggregate breakage of carbon black may lead to decreased conductivity. In order to ease the manufacturing of batteries other methods for stabilization are investigated.
The presented approach is the hetero-aggregation of a nanoparticulate silica on-to the aggregate structure of carbon black in spray flames. By achieving sintering bridges of the two distinct materials on the primary particle level the aggregate stability can be improved. However, the quantification and assessment of the mixing quality which is pivotal for the desired functional properties of the hetero-aggregates is still a major challenge.
The presentation will cover results of the multiscale structural characterization of hetero-aggregates consisting of carbon black and silica via a combination of small angle X-ray scattering (SAXS), analytical disc centrifugation (ADC) and transmission electron microscopy (TEM). Therein, the key influences of the experimental conditions on the aggregate structure are determined. Afterwards, a methodology to assess the mixing quality by analyzing TEM-EDX images is presented. In this context, the presentation covers how classic mixing theory needs to be adapted to be suitable for image analysis of nanoparticles. Furthermore, a metric which allows the comparison of hetero-aggregates of different composition and scale will be presented.

Content

Experimental and simulative studies on the structure formation of SiO2-PS heteroagglomerates

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Abstract

The development of tailor-made particle products with additional integrated functions is based on a mechanistic understanding of particle processes. Heteroagglomeration via gas phase processes is a method to combine existing particle properties or to create new functions by combining them. In this work, experimental and simulative approaches are combined to analyse the complex mechanisms of heteroagglomeration and to identify process-structure-property relationships.
Silicon dioxide and polystyrene model particles are used for the experimental preparation of the heteroagglomerates. First, the primary aggregates are produced separately using spray dryers. The aggregates are then agglomerated in a defined zone to produce heteroagglomerates. In order to investigate the influence of process parameters on the structural properties, the existing flow conditions in the process are systematically adjusted.
The use of coupled flow (CFD) and particle simulations (DEM) offers the possibility to systematically investigate the heteroagglomeration processes of submicron particles as a function of process parameters, dispersed particle properties and various particle interactions. The DEM takes into account DLVO theory, lubrication forces, Brownian motion. The adhesive forces acting in the simulation are calibrated and validated by AFM measurements. Structural analyses provide information on the coordination number, the number of heterocontacts and the radial structure of the heteroagglomerates.
The aim of the studies is to understand the complex relationships in the formation of heteroagglomerates and to determine the process-structure-property relationships. Among other things, this should contribute to the manufacture of tailor-made products with specific material properties.

Content

CFD Investigations of Multiphase Mixing of Heterogeneous Nanoparticles in an Opposed Jet Fluidized Bed

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Friedrich-Alexander-Universität Erlangen-Nürnberg (FAU)

Abstract

Hetero-aggregation is a process in which two or more distinct particles are mixed and blended together to form a new particle which has superior properties as compared to the original constituents. Such manufactured particles exhibit better performance in different industrial applications such as pharmaceuticals, synthetical organs, photochemical processes, dyes, energy devices etc. Having several merits, dry formulation technique (i.e., gas phase mixing) is adopted for the formulation of hetero – aggregates in which a high velocity jet is introduced into a closed domain, i.e., fluidized bed for intensive mixing and blending of particles. However, highly turbulent flow represents a complex system with high momentums caused by particle to particle and particle to gas collisions. Hence, it is necessary to explore multiphase behaviour and internal fluid dynamics of such systems for efficient outcomes. In the current study, an E-E multiphase CFD model has been implemented for the investigation of multiphase mixing and internal hydrodynamics of an opposed jet fluidized bed. Multiphase system comprises of three phases, i.e., air was considered as primary phase whereas TiO2 and ZrO2 particles were considered as the secondary phases. Particles were placed in the reactor domain at known depths and air jet was injected with the help of three nozzles mounted in the bottom and side walls. Preliminary findings showed that fluid dynamically different zones are formed in the domain such as mixing zone, stressing zone and reforming zone. Further investigations are being carried out for the parametric optimization with respect to different particle packing depths, particles sizes, jet injection positions and velocities.

Content

Mixing Mechanisms in Intersecting Spray Flames for Hetero-Aggregate Particle Production

Tobias Tabeling; Prof. Dr. Udo Fritsching
University of Bremen / Leibniz-Institute for Materials Engineering - IWT

Abstract

Nanoparticles may possess specific customizable characteristics, including precise size, composition and morphology. For producing hetero-aggregated nanoparticles the Double Flame Spray Pyrolysis (D-FSP) process can be utilized. Due to the inherent complexity of the D-FSP process, a thorough understanding of the mixing mechanisms between the two spray flames is essential.
The D-FSP setup employs two FSP spray nozzles. This configuration has been modeled and simulated using a hybrid RANS / LES approach in OpenFOAM. The individual spray flames are modeled with RANS while the mixing zone between the two spray flames is captured in detail using LES. A custom solver, specifically developed for simulating the FSP process, has been adapted and extended to validate the new turbulence modeling technique.
The mixing phenomena have been investigated in various D-FSP configurations varying the distance between the two nozzles, the distance between nozzles and mixing zone and the angle of the individual FSP nozzles. The findings indicate that external gas entrainment plays a pivotal role in the mixing process of the D-FSP, emphasizing the need for investigation the gas entrainment impact. From the simulation results the formation of particulate hetero-contacts is determined. A comprehensive understanding of the mixing mechanisms enables the precise design of hetero-contacts by tailoring mixing and sintering durations.
The financial support of this project through the German Research Foundation (DFG) within the priority program SPP 2289 „Hetero Aggregates“ is gratefully acknowledged. Furthermore, the authors would like to thank the NHR for computing time under the grant hbi00064.

Content

Transport, deposition and interaction of aerosol droplets and chips in drilling processes

Teresa Tonn MSc[1]; Lukas Schumski MSc[1]; Dr. Lizoel Buss; Dr. Anne Katharina Geppert[1]; Dr. Sölter Jens[1]; Prof. Dr. Bernhard Karpuschewski[1]; Prof. Dr. Udo Fritsching[1]
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Abstract

Cooling lubricants are used in machining processes; they reduce friction and thus frictional heat, while also contributing to heat dissipation (cooling-lubricating-transporting). In minimum quantity lubrication (MQL), the cooling lubricant is atomised in a gas phase and transported into the machining zone as an aerosol. The distribution of the cooling lubricant in the machining zone depends on the geometric boundary conditions, the drilling process control variables, and the cooling lubricant, gas and aerosol properties (droplet sizes and gas velocities).

This study investigates the distribution of cooling lubricant in the borehole bottom, with internal cooling lubricant aerosol supplied by the drilling tool, as a function of drilling parameters, cooling lubricant formulation, and aerosol properties during vibration-assisted drilling (VAD).

The Euler-Lagrange-Euler (ELE) simulation model includes the gas flow as an eulerian phase and the aerosol droplets as a discrete lagrangian phase (DPM). Coupled with an Euler model for film formation at the bottom of the borehole, the model demonstrates film formation of variable thickness right underneath the cooling channel, which would result in a cutting edge that is only partially lubricated.

In discontinuous drilling processes such as VAD, chip formation produces small particles shaped like segments of the borehole bottom rather than long spirals. The interaction between aerosol droplets and particles is verified by measuring lubricant residues on the particles. Furthermore, particle transport is investigated experimentally and through coupled CFD-DEM simulations, showing a dependency on the gas phase velocity.

Acknowledgement: Funded by DFG (FR 912/46-2) within SPP 2231 FluSimPro.

Content

Exploring the internal dynamics of resonant acoustic mixing using positron emission particle tracking

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Abstract

Blending composites like syntactics and resin-based systems conventionally occur through extended mixing in rotary drums or traditional blade-based mixers. After the blending process, the mixture is poured, molded into blocks, and subjected to subtractive machining. These procedures are time-intensive, generate waste, and pose potential hazards, especially when dealing with materials sensitive to energy. To address these challenges, we have explored the application of Resonant Acoustic Mixing (RAM), an innovative technology for blending powder/powder, powder/fluid, and fluid/fluid mixtures. So far, we have studied the two most fundamental variable parameters in RAM mixing – the fill height of the vessel and the strength with which it is vibrated. To test how the fill level and the vibrational acceleration to which it is exposed affect mixing efficiency, we have performed a design of experiments imaging a simple material, microcrystalline cellulose (MCC) within a RAM mixer using positron emission particle tracking (PEPT). We observed that there is a clear, monotonic, positive relationship between acceleration and mixing efficiency. However, while we observed that fill level has a clear influence on mixing efficiency, the form of this relationship is currently unclear. In the current study, we are focusing on the relationship between aspect ratio and mixing efficiency. Previous studies showed that aspect ratio (AR) influences mixing dynamics and heating, but no systematic study has been performed. To clarify the effect of aspect ratio on mixing quality and efficiency we decided to study this relationship further by using five different aspect ratios for mixing vessel as; 0.25, 0.5, 1, 2 and 4 with constant volume by using PEPT.

Polymer Suspensions under Uniaxial Extension – A Novel Way to Produce Core-Shell-Particles?

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Abstract

Microscale core-shell particles can combine properties of both phases and even release their impact at different timescales. The multitude of applications in the pharmaceutical, for example targeted drug delivery and release, and chemical industries, for example catalysts, shows the significance of these structures and underlines the need for new production routines. In the research presented, the focus lies on the bonding properties of the two phases to introduce these structures to new applications like selective laser sintering (SLS), an additive manufacturing process.
In the field of SLS microscale thermoplastic polymer powders are fused into 3D objects, by means of selective heating. The strength and other properties e.g. flame retardancy, of these components can be highly improved through the targeted use of (nano-)particles and fibres in core shell structures, since simple dry mixing is not sufficient to achieve the desired properties Novel powder production methods, such as filament extension atomization can be used to compound the particles directly into the powder during production.
In filament extension atomization, polymer filaments are continuously stretched at high extension rates until instabilities form and the filaments decay into droplets. At this point, particles can be used to create defined instabilities.
The moment of instabilities as well as the viscosity and particle concentration dependence of the droplet sizes formed were investigated in order to compare them with previous experiments with polymer solutions and to investigate the question of whether the number and size of the droplets can also be specifically adjusted in polymer melts in the same way as with polymer solutions.

Content

Comparative analysis of particle behaviour with different stirrer geometries in a capsule filling machine using DEM simulations

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Abstract

In the present study, a capsule filling machine with a powder chamber and stirrer located above a vacuum drum was analysed. The drum contains four lines, each with five bores, dosing powder into five capsules simultaneously. The stirrer ensures uniform distribution and constant filling of the capsules. It has been shown experimentally that the stirrer geometry has a significant influence on the powder behaviour in the chamber. Discrete Element Method (DEM) simulations were used to obtain detailed information about particle motion and mixing in the chamber, enabling a comprehensive comparison between different stirrer geometries. A lactose powder was considered, and due to its small particle size, coarse graining had to be applied. A calibration was performed using an auger dosing system to obtain the input parameters for the simulations. Validation experiments involved placing a tracer in the middle of the chamber, measuring axial tracer distribution after one minute of stirrer rotation using UV-Vis spectroscopy, image analysis, and simulations. Results showed similar powder bed profiles and tracer concentrations along the chamber, with reasonable agreement despite some differences. The calibration proved sufficient for comparative analysis of stirrer geometries and particle movement. The Lacey mixing index, total and net displacement of the particles, average particle velocities, and velocity fields were analysed. The simulations provided detailed information at the particle level, revealing how particles move and behave differently depending on the stirrer geometry. This information is highly valuable for the development of future stirrer designs, optimization of the filling technology, and understanding material behaviour.

Content

Online velocity estimation in cold gas spraying using acoustic measurements

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Abstract

Cold gas spraying (CS) is gaining increasing attention in the field of additive manufacturing due to its ability to utilize a broad variety of materials, such as metals, ceramics, and polymers. The bonding of solid particles (5-60 µm) to the surface is ensured by the high energy of impact. The impact velocity is a key factor determining bonding efficiency, where a critical velocity must be exceeded to achieve bonding, and plastic deformation. In this work, a process concept was developed to enable autonomous 3D additive manufacturing of complex geometries with high resolution. For process automation, the monitoring of the particle velocity as a process-decisive variable is of particular importance. Although different methods exist to measure particle velocity in cold gas spraying, such as High Speed Particle Image Velocimetry (H-PIV), the application of these methods in the manufacturing operation is often limited due to the small distance between the nozzle and the substrate. An alternative approach is the implementation of an non-optical measurement method, that allows for the online quantification of process parameters under harsh conditions. Due to the high velocity inside the jet and the high energy of impact, acoustic signals are emitted during the CS process. These acoustic signals contain information about particle velocity, which presents a new potential method for non-intrusive online estimation. A soft sensor based on audio signal analysis from a real cold gas spraying system was developed in this in this work, that allows for real-time velocity estimation. Despite the higher variance in the data compared to PIV methods, this acoustic approach still enables the determination of the bonding regime, allowing the online adaptation of process parameters.

Content

Data-driven Soft-Sensor for Real-Time Process Control in Stirred Media Mills

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Abstract

Optimizing the performance of wet stirred media mills remains a significant challenge due to the dynamic interplay between particle size, suspension rheology, and bead motion during fine grinding processes. Conventional models, based on static process assumptions, fail to capture the evolving conditions that occur as particle sizes decrease and suspension viscosity increases. As particle sizes decrease, the optimal stress energy required for effective comminution diminishes, whereas increasing viscosity demands higher energy inputs to maintain efficient bead dynamics and energy transfer. This work presents a framework for real-time process control, integrating data-driven modelling and advanced simulation techniques to address these dynamic challenges.
In the presented framework, a soft-sensor tracks particle size and viscosity for subsequent processing in a population balance model embedded within a nonlinear model predictive control architecture. AI-powered soft sensors provide continuous, real-time predictions, eliminating reliance on offline measurements. By correcting sound-based online data and integrating online mixer torque data, neural networks predict in real time more accurate PSD measures, comparable to optical measurements, as well as shear-rate-dependent viscosity data, not being measured online at all. Coupled computational fluid dynamics and discrete element method simulations, enhanced through machine learning, deliver high-resolution insights into bead motion and collision patterns, informing process optimization. Evolutionary algorithms further enable automated calibration and refinement of PB models to ensure accurate real-time predictions.
This research is funded by the Deutsche Forschungsgemeinschaft (DFG) under SPP 2364.

Content

Influence of filter cleaning frequency on the particle size distribution of granules in continuous wet granulation

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Abstract

Continuous twin-screw wet granulation efficiently produces pharmaceutical granules. In the QbCon 1 system, granules dry on a porous bed with temperature-controlled airflow and vibration for consistent processing. A filtration system prevents fine particle loss using ultrasonic cleaning and airflow reversal. This study aimed to assess filter cleaning effects on critical process parameters (CPPs) and critical quality attributes (CQAs), including residence time distribution (RTD), content uniformity, and particle size distribution (PSD).
The formulations were granulated with demineralized water as granulation liquid. To determine the RTD in a pulse experiment amaranth 85 E123 (BASF ChemTrade, Germany) was used as a tracer.The PSD was analysed using the Haver CPA (Haver & Boecker, Germany) and the Mastersizer 3000 (Malvern, United Kingdom).
Granule size fluctuations affect CPPs and CQAs, including RTD and content uniformity. Smaller particles tended to accumulate in the filter, prolonging residence time and risking non-uniformity, highlighting the importance of filter cleaning cycles and formulation properties for product quality.
German Research Foundation (DFG). SPP 2364, Grant number: 504702251.

Stochastic modeling of particle structures in spray fluidized bed agglomeration using methods from machine learning

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Abstract

Agglomeration is a fundamental industrial process for producing bulk materials. The effective properties of products made from bulk materials depend heavily on the distribution of agglomerate morphologies, e.g., size and shape. Thus, accurately characterizing and controlling these morphologies is essential to ensure high and consistent product quality. Spray fluidized bed agglomeration is a versatile technique in which a liquid binding agent is sprayed onto fluidized particles to form agglomerates. This talk introduces a pipeline for image-based inline agglomerate characterization and the prediction of their time-dependent multivariate morphology distributions within a spray fluidized bed process. The proposed framework considers three distinct morphological categories—primary particles, chain-shaped agglomerates, and raspberry-shaped agglomerates—based on size and shape descriptors derived from inline imaging done with Camsizer. A fast and robust random forest classifier [1] is trained for accurate and efficient categorization, making it suitable for inline measurements. Additionally, the fractions of these morphological classes and the two-dimensional distributions of their size and shape over time are modeled using low-parametric regressions and Archimedean copulas [2]. This approach not only enhances understanding of agglomerate formation but also enables predictions of aggregation time, necessary for achieving desired class fractions and morphology distributions.

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Coarse-Grained DEM-CFD Simulation of a Wet Bead Mill for Industrial Applications

Yuki Tsunazawa
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Abstract

For the grinding and dispersing of fine particles, wet bead mills are commonly employed in various industries including chemical, pharmaceutical, ceramic and mineral processing. To achieve efficient grinding and dispersing processes in wet bead mills, a deep understanding and appropriate control of bead particle behavior in mills are essential. As computer hardware continues to advance, numerical simulations—particularly the discrete element method (DEM) coupled with computational fluid dynamics (CFD)—have emerged as promising approaches. However, the existing DEM-CFD method faces significant challenges due to limitations in the number of computational particles despite the high demand for large-scale systems in industrial applications. To address these challenges, this study newly introduces a numerical model that integrates the coarse-grained DEM-CFD, an implicit algorithm for the drag force term, and a scalar field-based wall boundary for simulating wet bead mills. The proposed model successfully reproduces the macroscopic behavior of bead particles, as demonstrated through validation tests in a wet bead mill system. Specifically, a strong agreement is observed between the original particle system and the coarse-grained particle system in terms of particle location, particle velocity and total kinetic energy. Consequently, this study highlights the applicability of the proposed model for wet bead mill simulations and its potential contribution to numerical investigation aimed at optimizing industrial wet bead mill applications.

Content

Analysis of DEM-calendered anode microstructures for electronic and ionic conductivity

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Abstract

Within lithium-ion battery production, dedicating attention to all process steps along the fabrication chain and understanding their influence on the batteries' performance indicators is crucial for enhancing battery operation. Such key indicators include the electrode's electronic and ionic conductivity, which are both determined by its chemical and structural composition. The calendering process primarily determines the final microstructure of an electrode. By compacting the electrode-current collector composite, calendering contributes to the mechanical integrity of the cell and reduces the microstructure's porosity. An optimal calendering degree produces electrodes with enhanced electrical conductivity and mechanical stability at the same time as high ion mobility, making it a critical factor for later battery performance.
In this work, the calendering process of anode microstructures is simulated via the Discrete Element Method, and the resulting microstructures are analysed for electronic and ionic conductivity. The graph-ite anodes are modelled as particulate structures, where contact forces act between the particles and contact models determine the resulting particle deformations. While simulations with spherical active material particles have already been conducted, the densification behavior of non-spherical particles, such as those used in graphite anodes, remains unexplored. Therefore, non-spherical particles representing the actual shape of graphite are used in the calendering simulations and the subsequent structural analyses, yielding realistic results. A correlation between the degree of densification, forces acting on the particulate graphite anodes, and the final electrode performance is established and in good agreement with experimental values.

Content

Using Simulation, HPC and Machine Learning to virtual Optimize Rotary Dryer Operations for Enhanced Efficiency

Marcus Schierle
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Abstract

Rotary dryers play a critical role in industries such as mining and agriculture but are often challenged by high-energy consumption, equipment wear, and inconsistent drying performance. This study investigates the integration of discrete element methods (DEM), computational fluid dynamics (CFD), and machine learning to efficiently optimize both the design and operational conditions of rotary dryers. By leveraging the computational power of high-performance computing (HPC) systems, high-fidelity simulations provide detailed analyses of particle-fluid interactions, heat transfer mechanisms, and moisture evaporation dynamics.

Machine learning techniques, applied to the simulation-generated data, allow optimizing key parameters rapidly like rotational speed and airflow in a strategic manner. This method remarkably accelerates the optimization process and enables the implementation of multi-objective optimizations of both the equipment's physical geometry and process conditions, driving significant improvements in energy efficiency and wear reduction.

The synergy between HPC-enabled simulations and machine learning improves the decision-making process and supports the development of robust solutions for real-world industrial challenges. Key outcomes of this approach include actionable insights into geometry optimization, precise temperature control, and advanced predictive maintenance strategies. These innovations collectively deliver substantial reductions in operational costs and extended equipment lifespan, providing a scalable, industry-wide framework for sustainable process improvement.

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Innovative method evaluating permeability for tableting process improvement

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Abstract

Permeability has been observed to be a useful metric for tableting, correlated with the tensile strength of the produced tablet. Permeability is the ability of a powder to let the surrounding fluid (generally air) pass through the inter-particle voids of the powder bed. This ability of the air to pass through the powder and to be removed from the powder during compression highly influences the quantity of entrapped air at the end of the process. This small quantity of air inside the tablet can be responsible for a decrease in the tensile strength of the tablet, leading to possible breakage. Therefore, measuring permeability can prevent such issues by by an early identification of powder formulations with insufficient tensile strength.

In this work, a comparative analysis was conducted between the tablet properties and permeability for three different powder mixtures composed of an excipient (SuperTab® 11SD, SuperTab® 30GR, and SuperTab® 21AN, DFE Pharma, Germany) and a lubricant (Magnesium stearate, Peter Greven, Germany). The permeability was measured with an innovative tool combining tapped density measurement with a cell to measure permeability (GranuPack permeability, Granutools). While many methods only quantify permeability at a fixed packing fraction, this instrument can measure permeability at different densification states. Permeability values at various packing fractions and metrics related to the decrease in permeability with packing fraction were investigated for the three powders and compared to the tensile strength of the final tablet. The new insights gathered from this approach give a better understanding of the material properties and allow the development of improved formulations for better processability.

Content

Real-Time Dynamic Extinction Spectroscopy Sensor for Monitoring Precipitation and Crystallization in Phosphorus Recovery from Wastewater

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Abstract

In order to gain insight into the process dynamics and kinetics of the crystallization and precipitation of Magnesium Ammonium Phosphate (MAP) from wastewater, a sensor, based on Dynamic Extinction Spectroscopy (DES) [1, 2], has been adapted and implemented on a lab-scale crystallization reactor in the line of this work, enabling real-time analysis of the process. Based on physical principles, such as the Mie theory and Lambert-Beer law, the DES combines statistical and spectral extinction methods, allowing for accurate particle size and concentration measurements derived from transmission signal analysis. The sensor's construction and experimental validation using model materials, such as polystyrene spheres and MAP particles of known size distributions are presented. Furthermore, initial results from these experiments highlight the sensor's ability to capture dynamic changes in particle size and concentration, paving the way to gain critical insights into process parameters, such as pH and temperature. Insights from the sensor are to be integrated into macroscopic Population Balance Models (PBM) alongside microscopic parameters from DEM-CFD coupling. This combined approach aims to deepen understanding of nucleation, growth, attrition, and breakage dynamics, ultimately enabling the development of predictive models and strategies for enhanced process optimization.

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Content

The potential of microstructure diagnostics for particle analysis

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Abstract

The physical, chemical, and biological properties of micro- and nanoparticles depend significantly on the particle size, shape, and particle size distribution. In particular, the determination of the particle size distribution of powders is currently of high relevance in a variety of different fields such as medicine, electronics, energy, and environmental sciences in order to meet regulatory requirements.
By using advanced nano- and surface analytical technologies, it is possible to gain direct access to the previously mentioned particle properties. In the case of coated particles, statements on layer thicknesses, layer adhesion, interface properties and structural morphology are also possible. By means of energy dispersive X-ray spectroscopy (EDXS), the lateral element distribution in the particles can be determined allowing conclusions to be drawn about the homogeneity of the particle itself or the surrounding layer(s).
Various practically relevant examples (effect pigment, phosphor, UV-absorbing particles) are shown to illustrate the workflow from artifact-free sample preparation to characterization using high-end analytical methods. It is shown that micro- and nanostructure diagnostics, especially when operated at the resolution and detection limit of currently available analytical techniques, can only provide high-quality results if the samples meet the requirements of the various analysis methods. Therefore, sample preparation is a critical step in particle analysis. Moreover, it is emphasized that a combination of different analytical methods such as transmission electron microscopy (TEM) or time-of flight secondary ion mass spectrometry (ToF-SIMS) is beneficial, as these provide unsurpassed lateral resolution as well as high detection sensitivity.

Content

Advancing in Compaction Simulation: Key Insights into the Scale Transfer of Die Filling

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Abstract

In the early stages of tablet formulation development, compaction simulators provide a great deal of knowledge about the compression behavior and tablet ejection. However, the critical step of die filling, which determines the weight and therefore the content uniformity of the resulting tablets, is currently not accurately reflected in compaction simulation. Existing filling models [1] are currently only applicable to rotary presses, and the large constructional discrepancies limit their transferability to compaction simulators.
The aim of this study is to derive key factors for the rational scale transfer of die filling towards a laboratory scale rotary press. Model excipients of poor, medium and excellent flowability serve as surrogates for different degrees of challenging formulations. Differential pressure and permeability measurements provide insight into the impact of suction filling. Different feeding systems are used to investigate the constructional aspects that may represent dynamic conditions on rotary presses to a different extent. Extensive powder characterisation, including ring shear testing and dynamic angle of repose, aims towards finding suitable predictors for the filling efficiency on the compaction simulator. The derived hypotheses are tested and refined by comparative filling trials on a laboratory scale rotary press.
Ultimately, these extensive analyses provide the dataset and framework of knowledge for a filling model that enables compaction simulation to reflect the entire tableting process in early formulation and process design, streamlining the knowledge transfer from R&D to production.
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Simulation of lunar regolith particle motion behaviour during spacecraft landing with a supersonic multiphase solver

Tobias Laming[1]; Jannis Petersen[2]; Martin Propst[2]; Theodor Heutling[2]; Dr.-Ing. Christian Bach[2]; Bradley Craig[3]; Prof. Dr. Konstantinos Kontis; Dr.-Ing. Stefan Linke[4]; Prof. Dr.-Ing. Enrico Stoll[4]; Dr. Jouke Hijlkema[5]; Dr. Jeroen Van de Eynde[6]; Prof. Dr.-In
[1] TU Braunschweig; [2] TU Dresden ; [3] University of Glasgow; [4] TU Berlin; [5] ONERA, The French Aerospace Lab; [6] European Space Agency

Abstract

A joint initiative involving NASA, ESA and several other space agencies is facilitating a human return to the lunar surface. In order to establish infrastructure and a sustainable presence on the lunar surface, it is essential to minimise the dust emissions and turbulence caused by the spacecraft exhaust. Therefore, it is planned to use an in-situ manufactured lunar landing pad in order to avoid the emission of dust and the turbulence caused by the spacecraft exhaust. Given the extreme conditions, including reduced gravity, high vacuum, and the sharp particle morphology, a combination of experiments and simulations is employed to describe the interaction between the lunar landing pad and its environment. For the experimental framework, a combination of microscale and macroscale methods will be used to characterise the materials used. For the simulation framework, a combination of computational fluid dynamics (CFD), direct simulation Monte Carlo (DSMC), and discrete element method (DEM) is used. This study presents a simulation model to investigate the trajectories of swirled-up particles induced by the spacecraft. A supersonic solver for CFD-DEM coupling, together with a modified drag force model of DiFelice for high-speed flows was implemented in the coupled simulations. Particles with a diameter between 40 µm and 800 µm were used to account for the size-distribution found in lunar regolith. To reduce the computational effort, the lunar landing pad is represented by a simplified STL geometry. The effect of various conditions on the resulting particle trajectories is determined and presented in a parameter study. For comparison, an overview of the experimental results obtained from the determination of particle trajectories in a high vacuum chamber is provided.

Content

Time-extrapolation of CFD-DEM Simulations for Iron Ore Reduction

Dr. Daniel Queteschiner; Dr. Thomas Lichtenegger; Prof. Dr. Stefan Pirker
Johannes Kepler University

Abstract

Applying DEM or CFD-DEM models to industrial-scale systems such as blast furnaces or direct reduction shaft furnaces unveils the immense numerical costs of these simulation methods. To reduce the required computational resources it is inevitable to decrease the level of detail of the applied method.
One possible strategy is the usage of a coarse-grain (CG) approach for the discrete phase, lowering the computational demand by using coarser (pseudo) particles. However, this approach may fail to capture effects that inherently depend on particle size. To alleviate these deficiencies, we have proposed a multi-level coarse-grain (MLCG) model. In this model multiple concurrently simulated coarse-grain levels are coupled to adjust the resolution of the system as needed. The MLCG model can also be applied to fluid-particle systems using CFD-DEM. This procedure allows us to cover the typical spatial scales of industrial plants.
To fully picture industrial processes involving the reduction of iron ore, however, we also need to be able to run our simulations for the full process time. While the chemical reduction of the material takes several hours to complete, even a coarse-grained CFD-DEM simulation is limited by a time step in the range of microseconds since particle contacts need to be resolved. To close this gap, we apply (flow-based) recurrence CFD (rCFD) as time extrapolation method, where particles are transported on precomputed recurrent flow fields and particle contacts no longer need to be resolved. The applicability of this model set is demonstrated by the reduction of iron ore pellets at elevated temperatures in a direct reduction shaft.

Content

Synthesis of monomodal, magnetic interference pigments

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[3] Technische Hochschule Nürnberg Georg Simon Ohm/Universität Ulm

Abstract

Pigments have been used as colourants for a long time. Today, pigments are also used in the decorative, cosmetics and automotive sectors. Interference pigments, whose colour changes depending on the viewing angle, are also used in these applications. In this paper, a process is presented that, in contrast to existing processes, does not require additional cleaning steps and at the same time provides interference pigments with a high monomodality. For this purpose, the conventional stripping of the PVD material from a carrier substrate is combined with a micro-embossing process. To test the process, several multi-layer materials are deposited in the PVD process. Magnetism, colour and thickness are varied. The particles are examined using light and electron microscopy, the particle size distribution is determined using MountainsLab® software, the colour and colour flip are determined spectroscopically and the magnetism is determined using a vibrating sample magnetometer.

Content

Mechanochemical Synthesis of Urea-Gypsum-Cocrystals

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Technische Hochschule Nürnberg Georg Simon Ohm

Abstract

Urea can be processed mechanochemically in ball mills with calcium sulfate to form an ionic urea cocrystal cocrystal (URCASU) with a stoichiometric ratio of 4:1. Compared to pure urea, URCASU has a lower solubility, hygroscopicity, decomposition rate in fertilized soil and an increased melting point. This property profile makes the substance an attractive and environmentally friendly alternative to classic urea and nitrogen fertilizers.
So far, no detailed experiments have been carried out to examine energy considerations in mechanochemical production in more detail. In the present work, a Fritsch Pulverisette 7 planetary ball mill was used with all hydrate stages of calcium sulfate (dihydrate, hemihydrate and anhydrite) and urea as starting materials. For the energetic evaluation of the process, the speed, grinding media size, grinding media filling ratios and grinding duration were varied. For the quantitative and qualitative evaluation, particle sizes were measured using laser diffraction spectrometry, particle morphology using light and scanning electron microscopy, and the degree of conversion using X-ray diffractometry and Raman spectroscopy. As a reference, URCASU was prepared from a solution to quantify the degree of conversion. It was found that comparatively moderate conditions were sufficient for quantitative conversion of the dihydrate and hemihydrate (e.g. 1 mm grinding media, 20 % grinding media filling level, 250 rpm for 10 min), while for anhydrite even harsh conditions (5 mm grinding balls, 20% grinding ball filling level, 1000 rpm for 60 min) only partially led to success. The degree of comminution apparently had no significant influence on the degree of conversion, although more precise investigations will be carried out before the conference.

Content

Characterization and structure reconstruction of aggregates from two-dimensional scale

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Otto-von-Guericke-Universität Magdeburg

Abstract

In previous study, the correlation between 2D box-count Df and 3D power law Df of aggregates has been investigated. Using the 2D projection method, the morphological representation of aggregates can be derived from two-dimensional image data. While this method facilitates the interpretation of fundamental principles, a significant limitation lies in the fact that 2D image data can only be obtained offline using scanning electron microscopy (SEM). Furthermore, retrieving optical data from aggregates composed of small primary particles (submicron particles) presents additional challenges. To address the need for real-time monitoring and automated control, a novel method must be developed. This study aims to establish a new correlation between the 2D box-count Df and the 3D power law Df, enabling a reliable estimation of the fractal dimension by measuring the entire contour of aggregates, including parameters such as cross-sectional area and maximum/minimum object lengths, while integrating the aforementioned projection method. A particle-cluster modified polydisperse tunable sequential aggregation (MPTSA) model is utilized to generate a series of aggregates for this investigation. The results will be compared with optical measurements and previous studies for validation.

Content

Prediction of the number of heterocontacts in two-component systems

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Abstract

Agglomeration of diverse materials can greatly enhance product quality and improve their physical, chemical, and optical properties. A key factor affecting these properties is the number of heterocontacts, which represent the interactions between distinct material components within the system. Numerous examples of altered properties resulting from heteroagglomeration and their related applications are documented in the scientific literature. However, the number of heterocontacts can be regulated by adjusting the setup used to produce heteroagglomerates. For instance, in the double nozzle flame spray pyrolysis (DFSP) method, altering the nozzle angle and distance can yield distinct final products due to a variation in the number of heterocontacts. Here, two different setups are evaluated: one involving a homogeneous mixture of primary particles made of different materials (mixed system) and another with initially separated primary particles (separated system). A comparison of the average number of heterocontacts across various cluster groups reveals that the system with homogeneously mixed primary particles results in a higher number of heterocontacts.
Predicting the number of heterocontacts from PBE simulations necessitates a detailed analysis of the growth in the number of heterocontacts. Simulations are conducted for various agglomerate combinations, involving agglomerates with differing primary particle number and composition. The frequency of new heterocontact formation during collisions is observed to correlate with the composition of the interacting clusters. Leveraging this information, a method is devised to compute the average number of heterocontacts throughout the agglomeration process while solving the population balance equation.

Content

A novel approach to model the particle dynamics for the synthesis process of hetero-aggregates

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Abstract

Hetero-aggregates are composite particles formed from two or more dispersed phases. The mixing of dispersed particle streams (aerosols) is a common pathway for hetero-aggregate production. The mixing process is modeled using CFD, where the dispersed phases are described as scalar fields in the Eulerian frame, while the dynamics of particle aggregate formation are governed by the population balance equation (PBE).
Modeling hetero-aggregate formation involves navigating high-dimensional spaces related to particle properties, such as particle volumes, fractal dimensions, and variations in primary particle types (e.g., composition). Consequently, conventional sectional models become computationally expensive for coupled CFD-PBE simulations.
In this study, we introduce a novel PBE model for hetero-aggregate growth from two or more mixing particle streams. This model builds on the monodisperse approach by Jeong and Choi [1] and is integrated into a CFD framework designed for high-Schmidt-number flows using the Eulerian-Lagrangian decomposition (ELD) method [2]. The model is validated against the sectional model proposed by Shigeta and Watanabe [3,4] for a binary material system. The results demonstrate that the new model achieves comparable accuracy while significantly reducing algorithmic complexity. Its low computational cost makes it highly suitable for integration into coupled CFD-PBE simulations.
We gratefully acknowledge the financial support by DFG in the scope of SPP 2289 (project No. 462475304).
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Characterization of mixing and structure in nanoparticle hetero-aggregates using convolutional neural networks: 2D-projection versus 3D-reconstruction

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Abstract

Functional properties of nanomaterials depend strongly on the underlying structure and chemical composition. One example are hetero-aggregates from double-flame spray pyrolysis, in which nanoparticles of two different metal oxides are mixed.
Improvement of functional properties requires a characterisation of structure and mixing. In conventional scanning transmission electron microscopy (STEM), 2D-projections of the samples are acquired, while information about the third dimension is lost. This can be overcome by STEM tomography, where the 3D-structure is reconstructed from a series of projections. However, tomography is expensive with respect to acquisition and evaluation time. Hence, 3D-reconstruction can only be done for a limited number of aggregates.
To obtain statistically relevant results, information on many hetero-aggregates has to be gathered from either 2D- or 3D-data. This can be challenging in regions where many particles overlap in 2D-projection. Convolutional neural networks (CNNs) can outperform a manual measurement or classical object detection algorithms. The application of CNNs requires a training of the network first, hence many training images with known particle positions and material types are required. Here, we simulated realistic 2D- and 3D-data of virtual hetero-aggregates.
We evaluated the trained CNNs using both simulated and experimental data in regard to the number of particles, the mass fractions, particle size distributions, heterogeneous coordination and the fractal dimension. We show that for a measurement of mass fractions and for the characterization of mixing, the evaluation of less expensive 2D-projection data is sufficient, whereas for a measurement of the fractal dimension a 3D-reconstruction is required.

Content

Investigation of the nano particle formation mechanics in a hetero-aggregation process by desublimation and supersonic flow

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Abstract

The controlled production of heteroaggregates composed of titanium dioxide (TiO_2) and copper phthalocyanine (CuPc) is significant for numerous applications, such as photovoltaics [1]. This study introduces an innovative aerosol process that combines desublimation and mixing in a supersonic flow to synthesize TiO_2/CuPc heteroaggregates in a targeted manner. A CuPc-laden aerosol is generated by heating copper phthalocyanine in a nitrogen carrier flow within a high-temperature furnace until sublimation occurs. The resulting gas mixture is accelerated to supersonic speed in a Laval nozzle and directed into a temperature-controlled expansion chamber. The rapid cooling during expansion initiates the desublimation of CuPc. Simultaneously, a titanium dioxide-laden airflow is introduced into the recirculation zone of the nozzle flow, bringing TiO_2 seed particles into the supersaturation region of the expansion chamber. Depending on process parameters such as temperature, pressure, flow rate, and particle concentration, different mechanisms of hetero-aggregate formation can dominate. The flow and particle processes were analyzed in detail using computational fluid dynamics (CFD) simulations to understand the flow dynamics, particle distribution, and formation of supersaturation regions. The CFD results also provide a com¬prehensive understanding of how process parameters affect particle formation and growth dynamics, paving the way for op¬timized control strategies to achieve desired material properties and process efficiencies.

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Numerical Simulation and Validation of Electrospray Deposition Patterns

Georgios Koukougkelis; Prof. Dr.-Ing. Martin Sommerfeld

Abstract

Electrostatic spraying of liquids is used for the production of quasi-monodispersed droplets with sizes ranging from nanometers to micrometers. The liquid to be atomized is fed through an electrified capillary tube forming a jet that disintegrates into very fine charged droplets which are carried by the electrostatic field to a collection plate. Electrospray deposition (ESD) has emerged as a powerful technique for creating thin films and coatings with precisely controlled properties, offering significant advantages in applications ranging from electronics and biomedicine to materials science and energy.

Previous numerical studies of electrospray deposition have often simplified droplet transport by using approximations for electrostatic interactions, such as the line-of-charge method, or by neglecting crucial phenomena such as Coulombic fission. Neglecting Coulombic fission is especially limiting as a single micrometer-sized droplet can undergo multiple fission events, yielding thousands of deposited particles. This study aims to develop a Lagrangian particle tracking model that incorporates these essential processes to accurately predict spray behavior and deposition patterns. The model considers the influence of drag, gravity, and Coulomb forces on droplet trajectories and incorporates a full coupling between the flow field, electrostatic field, and droplet phase.

The accuracy of the developed solver was assessed by comparing its predictions with experimental results. To further explore the sensitivity of the deposition process to variations in operating conditions, additional simulations were performed by varying key input parameters such as carrier gas temperature, gas velocity, and nozzle potential.

Content

Determination of droplet size from wide-angle light scattering image data using convolutional neural networks and synthetic training data

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Abstract

Wide-angle light scattering (WALS) offers the possibility of a highly temporally and spatially resolved measurement of droplets in spray-based methods for nanoparticle synthesis. The size and spatial distribution of these droplets are crucial for understanding the synthesis process.
In [1], the authors presented a neural network-based method for estimating droplet sizes from WALS images. However, in order to train such a network, training data had to first be obtained using traditional methods, which require some manual labeling. Moreover, these traditional methods struggle with WALS images depicting scattering patterns from multiple droplets, which can introduce biases into the neural network training in cases where droplets are densely packed.
To address these challenges, we present a fully automated workflow that uses synthetic training data and an enhanced network architecture capable of predicting droplet sizes in images with multiple droplets. This approach not only eliminates the need for manual labeling but also enables the use of systematically varied training data and a more thorough investigation of architectural limitations, such as determining how many droplets can be effectively distinguished in a single image. The trained network was then evaluated on a large dataset of nearly 35000 experimentally measured WALS images.
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Tracking hetero-aggregate formation in jet-based tube mixing via optical in situ detection

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Abstract

We present the application of a novel laser-optical in situ method for detecting gas-borne hetero-aggregates and distinguishing their individual components. The method employs a pump-probe laser detection scheme and is implemented in a jet-based tube-mixing experiment. Aerosolized particles are introduced via two dry-powder brush seeders, utilizing a model particulate system comprising carbon black (CB) and ZnO:Zn. The particle streams are injected into the mixing tube through two nozzles arranged at a 45° angle relative to the horizontal axis. The individual components can be distinguished by analyzing hetero-contact occurrences, wherein IR-absorbing CB particles are selectively heated upon pump(IR) excitation, while ZnO:Zn particles, serving as thermographic markers, exhibit temperature-sensitive luminescence upon probe(UV) excitation. A temperature-induced spectral red-shift of the centroid of the ZnO:Zn luminescence is observed exclusively in hetero-aggregates, indicating heat transfer from CB to ZnO:Zn upon thermal contact. The mixing process is investigated optically at five downstream positions over a total distance of 2100 mm from the mixing nozzles. The mass flow rates of CB and ZnO:Zn are adjusted to achieve a CB fraction of 5.4 wt.% of the total aerosol mass flow. Optical in situ detection reveals a higher occurrence of hetero-contacts in the near downstream region, while fewer hetero-contacts occur at the nozzle intersection. Complementary FMPS measurements at the same positions provide CB number concentrations, enabling the assessment of particle losses due to hetero-contact formation and their correlation with the spectral signal.

Content

Tailored hetero-aggregates from gas phase synthesis for heterogeneously catalyzed transient reactions

Dr Xiao-Xue Wang; Jakob Stahl; Max Gäßler; Franz Braun; Prof. Dr. Robert Güttel; Prof. Dr. Lutz Mädler PhD

Abstract

Hetero-contacts (HCs), defined as the interaction loci between two distinct solid materials, play a pivotal role in heterogeneous catalysis. Often manifested as metal-support interactions, HCs significantly influence the materials functions of metal nanoparticles by introducing novel interface phenomena. Understanding and controlling HCs in solid catalysts offer opportunities to tailor and enhance the catalytic performance significantly. In this study, catalyst materials with well-defined HCs are synthesized via Double Flame Spray Pyrolysis (DFSP), while Periodic Transient Kinetics (PTK) provides transient molar flow rates of reactive gaseous species. DFSP, as a robust single-step gas-phase synthesis method for the controlled formation of HCs, enables the mixing and hetero-aggregation of primary particles under tightly regulated conditions, including in situ heat treatment, which allows for the systematic adjustment of material interfaces. Parameters such as flow rates, flame distances and angles directly tune the number of HCs, primary particle size, and the chemical nature of catalytic interfaces. The mixing on hetero-aggregate level is quantified by CNN evaluations of HAADF-STEM images. To investigate the impact of hetero-aggregate features on catalytic performance, PTK method is applied to CO/CO_2 hydrogenation into CH_4. By periodically switching feed gas compositions and measuring outlet gas composition with high temporal resolution, PTK correlates the transient response of individual gaseous probe molecules to the chemical nature of the solid catalyst. Comparative analysis of experimental transient data and steady-state simulations revealed that reactant spillover across HCs between active and support materials is critical to species-resolved transient responses.

Content

Experimental study on the shape-dependent mixing behavior of binary mixtures

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Technical University of Munich

Abstract

Mixing of different granular materials is a critical step in many processes, especially in the food and pharmaceutical industries. Unfortunately, particle mixing is poorly understood, probably due to the complexity of determining the effects of complex particle shapes on mixing behavior.
More realistic random shapes are produced by generating meshes close to defined shape factors. This is achieved by optimizing spherical harmonic magnitudes. Using this type of optimization approach, the shape factors, i.e. convexity or aspect ratio, can be sampled independently and incrementally.
In our previous work, we identified particle shape combinations that showed different mixing behavior than the average of the Lacey mixing index trajectories. From this database, promising particle shape combinations were selected and then printed using a resin 3D printer. The printed particles are then placed in a rotating drum mixer in a 50:50 mass ratio. The Lacey mixing index is assessed by image analysis and verified by sampling from the drum mixer.
The mixing of the printed shapes results in a different evolution of the Lacey mixing index. Looking at the mixing of the two shapes, there is a tendency for a higher mixing rate (close to the curve for mixing shape I with itself) and a lower final mixed state (close to the curve for mixing shape II with itself). These results are in qualitative agreement with our simulations carried out previously. The outcomes are highly promising, demonstrating that the proposed method can generate controllable, realistic shapes while serving as a valuable tool to elucidate the effects of complex shapes on the poorly understood mixing behavior of binary mixtures.

Content

Suspension application on heated, structured surfaces for Smart-Alloying purposes

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Abstract

The demand for functionally graded materials has risen dramatically during the last decades. An effective way to fabricate them are additive manufacturing processes, and especially the Smart-Alloying concept. Smart-Alloying combines the conventional build-up process of laser powder bed fusion (PBF-LB/M) with a drop-on-demand (DoD) system for the targeted application of alloy suspensions (solvent + alloying element particles). This enables the local modification of chemical composition and micro structure and thus local alterations of the mechanical and corrosion properties of the metallic base material. The quality of the re-alloying process strongly depends on the precise distribution of the alloy suspension on the base material, i.e., on the droplet application itself. The latter includes the complex interplay of liquid properties, impact conditions and surface properties. This work focuses on understanding the dropwise deposition of alloy suspensions under PBF-LB/M process-like conditions. First, an experimental test rig emulating the drop-on-demand process during Smart-Alloying is being designed outside of the PBF-L/M system. This ensures that valuable PBF-LB/M machine times are not blocked and that the influence of individual parameters influencing the droplet-surface interaction, e.g., the properties of the suspension, the surface temperature and the surface structure, can be investigated separately. Therefore, the test rig will facilitate multi-perspective high-speed imaging. Here, we will present a proof-of-concept for the test rig and the results of a measurement campaign targeting the effect of surface temperature on the droplet-surface interaction. Specifically, we will characterize the impact outcome in regime maps and analyze the droplet spreading behavior.

Content

Suspension flow and dispersibility of soft swollen particles: Physically correct determination of the disperse phase volume

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University of Hohenheim

Abstract

In recent years a number of techniques have been developed to determine the swelling of particles [1]. These techniques allow to access the phase volume of the disperse phase which is of key importance to the flow behavior of respective suspensions and, if the dynamics of swelling are accessed as well, of the dispersibility of the analyzed powder. Examples of strongly swelling particles include proteins and biopolymers.
The difficulty in using the measured values is associated which the definition of swelling which inherently requires the determination of a phase boundary between particle and fluid. This is apparent in the case of image analysis as measurement technique where typically a threshold in gray scale defines this border. However, for the use in a physical model this border cannot be defined arbitrarily but needs to fit the mechanical reality. This has been studied empirically by correlating different measurement techniques to determine particle swelling and kinetics of swelling with the caused effect in suspension flow and dispersibility i.e. capillary rise.
The swelling of three different biopolymer particle systems (Soy protein, Pea protein, Gelatin) was accessed by Particle size analysis (absolute and relative), Image analysis, T2-NMR and gravimetrically. The first three of which also allow to determine the swelling kinetics.
The suspension flow was analyzed in the dilute form where particle-particle interactions can be neglected and at higher concentrations where the maximum packing density has an important influence. Dispersibility was accessed by capillary rise measurements.

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Novel green fibrous filter media for high-efficiency air filtration

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Abstract

Fibrous filtration materials are widely used in wastewater treatment, gas adsorption and separation, and particulate matter removal. Still, the eco-friendliness and stability of the filtration materials need to be further improved. Basalt fiber is a kind of inorganic nonmetallic fiber material with high performance prepared by crushing, melting, drawing, and coating with sizing, using natural basalt ore as raw material, which has excellent thermal and chemical stability [1]. This paper mainly introduces the fabrication of a novel fibrous composite based on nanocellulose fiber and micrometer basalt fiber and its application in air filtration. The all-natural fibrous composite filter was constructed using a wet method. Structural regulation for fine particle removal could obtain a novel high-efficiency filter material with a hierarchical structure (PM0.3 filtration efficiency up to 99.99%). In addition, surface modification engineering can also effectively regulate the electron gain and loss ability for composite filters, thus obtaining high electroactive fiber filters, and further improving filtration efficiency through synergy electrostatic adsorption. At the same time, by reconstructing the microstructure of fiber composite filter by simulation, the motion state of particulate matter can be visualized, thus ready to reveal the influence mechanism of parameters in the process of air filtration, and optimization strategies can be proposed.

Numerical Analysis of Fine Particle Impaction and Deposition on a Single Fibre and a Fibre Matrix in a Hot Gas Environment

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Abstract

In the present study the effect of thermophoresis on particle separation will be analysed by Lagrangian particle tracking for two model geometries. A single fibre with different diameter exposed to a hot gas laden with fine particles. The fibre temperature may be manipulated in order to modify the direction and strength of the thermophoretic force. The gas flow is considered to be laminar and the fibre Reynolds number is additionally varied through the inlet velocity. The separation efficiency of the single fibre is determined from the particles sticking to the fibre, normalised by the injected number over the projected cross-section of the fibre. The result for a single fibre is plotted versus the Stokes number for the different conditions. In the case of a cold fibre thermophoresis improves fine particle separation.
The second geometry is a fibre matrix with 4 cross-wise layers of fibres with fixed diameter (Sommerfeld et al. 2021). The gap between the layers is one fibre diameter and the gap between the fibres in each layer is two fibre diameters. The porosity of the fibre structure is 0.86. Again, fibre diameter and inflow velocity was varied. For a cooled fibre matrix, small sub-micron particles are mainly collected on the first fibre layer due to thermophoresis and for inertial particles of around one micron collection occurs also on the downstream fibre layers.
Particle deposition on the fibres is decided using an energy-based model (Sommerfeld 2022), which yields a critical particle impact velocity below which deposition occurs, otherwise particles will rebound according to the specified restitution ratio and friction coefficient (i.e., hard-sphere model). The parameters in the deposition model depend on the material properties of particles and wall.

Content

Viscoelastic phenomena at the receding three phase contact region during final stage of solid liquid filter cake formation

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Abstract

The separation of fine solid particles from a liquid is often accomplished by cake filtration. In the course of cake formation, this cake represents an increasing flow resistance. Thus, the final stage of cake formation is either particularly time consuming (in constant pressure mode) or energy intensive (in constant filtrate flux mode). This situation worsens when particle segregation effects by, e.g., superimposed sedimentation, result in a higher concentration of finer particles. As a consequence, the filter cake properties deteriorate even to the extent that the cake becomes almost impermeable and the supernatant fluid is removed very slowly.

The removal of the supernatant liquid may not occur homogeneously across the filter cake surface when it builds up non-uniformly, causing some filter cake surface patches to be still covered with the suspension while others are not. When the suspension is not provided evenly, the filter cake can form a non-symmetric tilted surface. Such a situation becomes even more pronounced when the suspension exhibits fast filtering and/or fast settling properties where the suspension has insufficient time to equilibrate evenly across the surface. Some filter cakes develop an uneven surface as a consequence of an inherently uneven cake formation process or cake shrinkage. Cake shrinkage occurs after the supernatant liquid has vanished and it may start off preferentially at locations where the cake exhibits heterogeneities. Along with horizontal cake shrinkage, cake cracking and cake dishing may occur.

Generally, at the end of filter cake formation the supernatant suspension is about to vanish and the continuity of the suspension layer breaks down locally. The suspension film forms three phase contact lines between the already formed filter cake, the suspension and the gas phase. In the vicinity of the contact line, the already formed filter cake expands to ease surface tension. The suspension film recedes vertically by filtration accompanied by a lateral movement of the film. This results in a wave of expanded cake that is subsequently densified again as a viscoelastic phenomenon.

Three markedly different aqueous suspensions, i.e., limestone, cellulose, and talcum, were filtered in a laboratory-scale Nutsche filter according to VDI guideline 2627. In comparison a cake was formed by evaporating the liquid thermally below the boiling point temperature. The interface of the suspension and the resulting filter cake was recorded by a camera to identify the moment when the optical reflection properties – shiny to opaque – changed, identifying the moment the supernatant liquid vanishes. Moreover, a spot of the surface was optically followed by a point laser system identifying the axial distance to the gas interface by diffuse reflection.

Content

The moment when the filter cake was just solidified but yet fully saturated with liquid correlated well with a drastically reduced random data scatter.

When the laser spot met the receding liquid front of the suspension film, i.e., three phase contact line, a distinct scatter of the laser signal was measured that insinuated substantial – actually unrealistically high – axial distance changes but instead can be attributed to the inclination of the reflecting surface. Such an extreme excursion of the laser signal was followed by a short period of lower more random scatter indicating that the surface was still fluid-like. Afterwards the laser signal provided a very stable signal typical for a solidified and immobile surface.

A suspension meniscus is located along the cake and cylindrical wall. When the laser spot is located in this area, a distinct scatter is found when the supernatant liquid is retracting radially from the center towards the meniscus. Actually the meniscus is found to vanish last. In the more central part, an apparent retracting front was only identified when the cake contained disrupting elements from which the film ruptured and retracted from there, or it had an inclined surface. When the film remained connected until it vanished solely by liquid removal (evaporation or filtering), no distinct scattering was observed.

Depending on the kind of suspension and operating conditions, the characteristic times of the receding suspension and compaction of the locally expanded filter cake were identified in the range of approximately 0.2 and 2 seconds, respectively. This time span was obtained between the onset of the distinct scatter until it vanished again. The magnitude and pattern of this scatter depended upon the geometry, i.e., the direction of the receding front in conjunction with the direction where the diffuse signal was picked up. Moreover, also the optical properties of the constituents, i.e., filter cake and suspension had an influence. The speed of the receding front also had an impact on the signal as well as the curvature of the cake and liquid. The subsequent filter cake solidifying period was in the range of up to around 15 seconds, i.e., one order of magnitude longer characteristic time.

Keywords: cake filtration, laser distance measurement, viscoelastic filter cake

Powder Photoemission-Current-Spectroscopy at Atmospheric Pressure

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Abstract

In this contribution a new setup for measuring the work function of metal powders, called powder photoemission current spectroscopy (PPCS) at atmospheric pressure is presented. There is a whole range of photoemission spectrometers at atmospheric pressure, which can basically be divided into two groups. On the one hand, the photoelectrons are transferred through a membrane from the atmospheric range via a skimmer system into a classic UHV-UPS/XPS device, but this is cost-intensive and complex. On the other hand, the emitted photoelectrons are detected by a charge multiplier or analyzed using a Kelvin probe after they have been attached to oxygen molecules. The setup presented here, which works with different gas compositions under atmospheric pressure and at room temperature, is based on the measurement of the photocurrent with a simple electrometer. It was developed for the online analysis of the oxidation state of metal powders used in oxygen-free production. All processes take place in a controlled environment at extremely low oxygen levels. The PPCS method has many advantages. For example, it is fast, safe, cheaper than UHV processes (such as UPS and XPS) and easy to use. It is also compatible with small amounts of powder (≈ 1 mg). In this work, fundamental questions about the technique were investigated such as particle size dependency, Child-Langmuir and Schottky effects.
The technique has been applied to several metal powders and metallic plates. Besides the work function also the photothreshold was determined.

Content

Hetero-aggregates produced by bipolar electrospray: particle characterization via UV-Vis absorption spectroscopy and wide-angle light scattering (WALS)

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Abstract

Heteroparticles, formed by two materials in contact, offer a wide range of novel applications, ranging from energy storage over drug delivery to sensing. A promising technique for the manufacturing of such hetero-particles with defined properties is the bipolar electrospray, as it enables the production of a variety of material combinations and morphologies. Yet the relations between the hetero-contact and the functional properties of the particles are barely understood due to insufficient characterization of the contact properties. Thus, appropriate measurement techniques are necessary in this context.
UV-Vis absorption spectroscopy (AS), based on measuring the attenuation coefficient of the nanoparticles, is particularly suitable to obtain insights into the optical properties. However, to derive parameters of interest, such as the absorption coefficient and the optical band gap energy, the measured extinction coefficient has to be corrected for the scattering influence. To this end, a setup consisting of UV-Vis AS combined with an integrating sphere to measure the wavelength dependent scattering cross section is presented.
To investigate a correlation between the absorption properties obtained by UV-Vis AS and the size and morphological properties of the particles, wide-angle light scattering (WALS) is employed additionally, allowing to determine morphological parameters from highly resolved scattering data. For a sophisticated evaluation of UV-Vis AS and scattering data from the hetero-particles simulations with discrete dipole approximation (DDA) are performed allowing to gain deeper insight into the hetero-contact by a combination of the two techniques.
The work was supported by the German Research Foundation (DFG) - 462396181.

Content

Operando dynamic light scattering of ultrasonic dispersion of nanoparticles

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Abstract

Ultrasonication is a frequently used, relatively simple, and rather inexpensive method to homogenize colloidal dispersions of nanoparticles. It is often the first step in processing powder-based colloids. The goal of this process is to disrupt existing agglomerates generating smaller particles, by this improving homogeneity and stability of the dispersions. For sufficiently high energy dissipated during ultrasonication, however, structural characteristics as well as physicochemical properties of the particles are changed. This may lead to undesired growth via sintering or decreased colloidal stability by modifications of the surface chemistry. Such ‘overprocessing’, should be avoided as it may affect the applicability of the nanoparticle dispersions. However, the effect of ‘overprocessing’ is usually only observed by ex situ characterization after finishing the sonication process.
Herein, we present operando investigations of nanoparticle dispersions during ultrasonication. Combining ultrasound (US)-assisted dispersing with dynamic light scattering (DLS), it is possible to continuously follow the development of the colloids with real-time resolution. These operando US-DLS measurements provide access to particle dynamics and changes of the particle size during the sonication process. The (transient) state of deagglomeration becomes directly accessible, without the need for interrupting the process for sample collection and ex situ characterization. The effect of varying sonication and suspension properties will be discussed.

This operando approach will reduce the potential of ‘overprocessing’, and allows for individual optimized processing of colloids.

Content

Visualizing Wetting Dynamics in Powder Beds using Terahertz Time-Domain Spectroscopy

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Abstract

Powder reconstitution is an ensemble of physicochemical phenomena that govern the entire solubilization mechanism. The first step of powder reconstitution, known as wetting, refers to the displacement of a fluid by another immiscible liquid. The knowledge and control of the wetting mechanisms are highly desirable, due to the high impact on the entire reconstitution process. Traditional methods for studying wetting include the sessile drop, Wilhelmy plate, and Washburn methods. These measurements are often used to calculate the surface free energy of the solid of interest, which dictates the wettability of a solid and, in turn, the liquid ingress in a powder bed.

This work will introduce a novel method for studying powder wetting. Using a reflection Terahertz Time-Domain Spectroscopy (THz-TDS) probe, the liquid front can be tracked during wetting. The method has been used extensively to study water ingress kinetics in compacts, but no studies have been published regarding loosely packed powders.

In the presented study the water ingress in lactose monohydrate is investigated. A custom-made sample holder setup was used, allowing THz-TDS measurement during liquid penetration due to capillary forces in the powder bed. Upon contact with the water, measurements show the formation of a front, which progressively ingresses into the powder bed over time, and slows down towards the surface.

The suitability of THz-TDS to investigate wetting and associated phenomena in porous powder beds is demonstrated. The method could prove valuable in improving reconstitution processes in the pharmaceutical and food industries through an increased understanding of the wetting step and subsequently occur-ring phenomena (swelling, collapse, sinking).

Content

Experimental Analysis of Triboelectric Behaviour of Cellulose and Lactose-Based Dry Powder Inhaler Formulations

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Abstract

Electrostatic charge, which often results from triboelectrification, plays a vital role in the handling of pharmaceutical powders. This phenomenon significantly impacts manufacturing efficiency and safety standards.Dry powder inhaler (DPI) formulations experience electrostatic charge during inhalation due to various mechanisms affecting particle segregation and dispersion. These mechanisms include entrainment in the gas flow, wall impacts, and the shear stresses generated during the process. DPI formulations typically contain a mixture of active pharmaceutical ingredients (APIs) and carrier particles, which significantly impacts powder delivery efficacy and stability. Given the complex nature of static electrification, additional studies are required to deepen our understanding of electrostatic charging in DPI formulations. This study investigates charge transfer in APIs under shear stress and their interaction with surfaces used in DPIs. The magnitude and polarity of charge in cellulose and lactose-based formulations are measured during wall friction testing using an Anton Paar powder shear cell. The effect of wall friction on tribocharging of the formulation is investigated by applying normal stress to the wall shear test apparatus and measuring its electrostatic charge using a Faraday cup. A custom shear cell was developed using insulating materials to prevent charge migration during the experiments. Further studies also explored tribocharging phenomena related to wall impact, specifically how electrostatic charging occurs from repeated collisions between API particles and DPI walls during inhalation. Through this investigation, we aim to enhance our understanding of charge dynamics in DPIs and provide valuable information about their performance and efficacy.

Content

Effect of Surface Functional Groups on Triboelectrification of Glass Beads

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Abstract

Pneumatic conveying of powders and grains often leads to significant electrostatic charge accumulation through triboelectrification, which can result in various processing challenges and, in extreme cases, pose risks of fire and explosion. The charge transfer process is influenced by the surface properties of the particles, which are, in turn, determined by their surface chemical functional groups and contact dynamics.
To investigate the role of polar and non-polar groups in charge transfer, glass beads were functionalized using acid washing (to render them highly hydrophilic) and silanization (to render them highly hydrophobic). These functionalized systems were further coated with aerosils (silica nanoparticles) to study their effect on modifying the charge transfer behaviour. Triboelectrification was induced using a cyclone, where particle impact and sliding brought the charge level close to equilibrium, as measured immediately after particles flowed into a Faraday cage. In this presentation, experimental results are reported, shedding light on the influence of surface treatments on triboelectrification.
The experimental data are then compared with numerical simulations using CFD-DEM, which incorporate experimental data on single-particle charge transfer. This approach enables the prediction of charge transfer behaviour based on the surface functional groups, providing a deeper understanding of the mechanisms at play.

Content

Gasification Behaviour of Biochar in the Chemical Looping Process

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Abstract

Chemical looping combustion (CLC) offers a promising approach for CO2 capture. In CLC, an oxygen carrier material (OC), is looped between two interconnected fluidized bed reactors and repeatedly oxidized in the air reactor using ambient air and reduced in the fuel reactor while converting the used feedstock. Since no nitrogen ever enters the fuel reactor, the flue gas consists only of CO2 and water, so no major scrubbing of the exhaust gas is required to realize carbon capture. This inherent CO2 capture capability is the key advantage of CLC over other CCUS technologies.
The fuel reactor is fluidized by CO2 and steam to gasify the biocoke remaining after the initial devolatilization. The accurate description of the kinetic behavior of the remaining biocoke during this in-situ gasification CLC process (iG-CLC) is crucial for the understanding of the process and the subsequent modeling of the fuel reactor.
Spouted beds, with a central gas inlet instead of a full-surface inlet, create a spout in the reactor center, enabling a circular motion of particles. This design enhances gas-solids contact and fluidizes hard-to-fluidize materials effectively.
After demonstrating improved performance under spouted bed conditions for oxygen carrier aided devolatilization of woody biomass, this work focuses on the gasification behavior of the remaining biocoke. In addition to the influence of the fluidization type, relevant influences such as temperature and particle size distribution of the char were investigated. Experiments were performed in a lab-scale batch reactor both with inert (sand) and reactive (OC) bed material. Both cases were studied separately with CO2 and steam to compare the reaction paths regarding their kinetics, resulting in a total of 96 parameter combinations.

Content

Neutron Imaging of Dynamic Microwave Freeze-Drying of bulk material

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Abstract

The gentle yet cost-efficient drying of sensitive products in the food and pharmaceutical industries is becoming increasingly important. However, the freeze-drying process is very time-consuming and costly, which is due to poor mass and heat transfer. Therefore, a microwave freeze-drying process was developed, in which the bulk material is continuously mixed in a rotating drum during drying.
To determine the influence of the drum's rotational speed on the drying process without interference, the drying process was observed using in-situ neutron imaging at the Centre for Energy Research in Budapest. This imaging technique allows for monitoring non-invasively not only the decreasing water content of the sample but also the changes in particle bulk. For this purpose, a dryer suitable for the requirements of microwave freeze-drying and neutron imaging was first developed, and then the drying process was imaged for different particle sizes and rotational speeds. These experiments enabled the non-invasive determination of the characteristics of the mixed particle bulk during the process.

Transformation of Pharmaceutical Manufacturing of Drug Substances with the Continuous Vacuum Screw Filter (CVSF)

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Abstract

Approximately 90 % of all active pharmaceutical ingredients (APIs) are obtained in crystalline form. Due to the increasing demand for continuous small-scale manufacturing processes (250 - 1000 kg/a), the development of continuous small-scale manufacturing equipment for the realization of a continuous crystal process chain (CPC), comprising of continuous particle generation and isolation is required. The patented modular Continuous Vacuum Screw Filter (CVSF) combines the unit operations filtration, washing, and drying in one flexible apparatus. The CVSF consists of a variable number of tubular modules connected via flanges and a screw. The modular setup enables a flexible process design by combining modules for filtration, washing, and drying. The rotating screw provides a gentle axial transport of the drug particles through the apparatus due to low rotational screw speeds of around 1 rpm, ensuring the preservation of particle specifications during processing. The CVSF is continuously fed with crystal suspension and provides continuous filtration, washing and drying.
In a continuous CPC, the particle isolation step follows the preceding particle generation via crystallization. Hence, the CVSF, realizing the continuous particle isolation, must be able to handle varying suspension flow rates and solid loadings, predefined by the preceding crystallization process. In this contribution, we will show how flexible the CVSF can be adapted for varying suspension flow rates or solid loadings, respectively. Therefore, different screw designs enable different residence times at optimal conditions. In addition, we focus on an optimal washing performance reducing waste throughout the process. The results are shown for a low-cost model substance system, namely L-alanine/water.

Content

Ferrimagnetic nano susceptors for sustainable adhesive technologies using high-frequency induction heating

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Abstract

As industries strive for sustainability, there is an increasing demand for innovative adhesive solutions that enable fast curing and debonding on demand. Ferrimagnetic nano susceptors for high-frequency induction heating (HFI) represent a transformative approach in this context. These nano susceptors can be rapidly heated using HFI, achieving heating rates of up to 1500 K/s to trigger rapid curing or debonding processes.(1) HFI provides targeted energy input, significantly reducing overall energy consumption during adhesive processing. The ability to control temperature precisely and spatially allows for tailored adhesive properties that meet the evolving needs of sustainable product development.(2)
Using ferrimagnetic nano-susceptors, we implemented debonding on demand for thermoplastic adhesives, allowing for reversible bonding strategies. The nano susceptors can be customized to optimize the performance of various adhesive formulations. For structurally bonded thermosets, complex particles comprising both nano susceptors and blowing agents can be utilized.(3) This innovative approach enables controlled debonding processes while maintaining structural integrity during use.
In summary, the integration of HFI with ferrimagnetic nano-susceptors and complex particles offers fast curing and debonding on demand with high heating rates and spatially resolved control. This technology thus enhances product performance while aligning with sustainability principles. Further research in this area is essential to realize the full potential of HFI in creating innovative adhesive solutions for a sustainable future.
(1) doi: 10.1016/j.jmmm.2019.165350.
(2) doi: 10.1016/j.jmmm.2024.172042; 10.1002/adem.202400744.
(3) patent submitted.

Interfacial influence of filler particles on polymer-based electrolytes for solid-state lithium batteries

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Abstract

Solid-state lithium batteries (SSLBs) based on solid polymer electrolytes feature improved safety and high specific energy. However, the low ionic conductivity of polymer electrolytes at room temperature (RT) is a major limiting factor for operation. Hybrid solid electrolytes (HSEs) consisting of filler particles incorporated into an polymer matrix show improved properties and increased conductivity at RT.
In this contribution, novel concepts and investigations on the effect of particles on improving the properties of polymer electrolytes are presented, emphasizing the importance of the particle-matrix interaction for the enhancement of ionic conductivity.
In one study, the effects of passive ZrO2 and active Li7La3Zr2O12 (LLZO) particles on a standard polyethylene oxide (PEO) electrolyte were compared. The particle surface was functionalized with a silane ligand prior to their integration into the polymer. Thereby, even though being non-ion conductive themselves, the ZrO2 fillers enhanced the ionic conductivity at RT up to 6.66·10-4 S cm-1 by reducing the crystallinity of PEO (compared to 9.25·10-6 S cm-1 for the PEO), while the functionalized LLZO fillers did not improve the conductivity, due to a LiCO3 passivation layer1.
In a subsequent study, the effect of LLZO particles subjected to a previous surface functionalization step involving the removal of LiCO3 on a cross-linked copolymer HSE was investigated2. While the cross-linked nature of the copolymer prevents its crystallization at low temperatures, the addition of functionalized LLZO resulted in a promising improvement in the ionic conductivity up to 1.14·10-3 S cm-1. This enabled the operation of a Li-S SSLB at RT and promises a general strategy for the design of HSEs for RT practical applications of SSLBs

Content

Integration of La0.8Sr0.2CoO3 supraparticles as structured anode materials for enhanced oxygen evolution reaction in alkaline conditions

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Abstract

Noble metal electrocatalysts are the most efficient for oxygen evolution reaction, but their scarcity and stability limit their applications. Consequently, future research has focused on developing non-noble metal electrocatalysts such as perovskites. Nonetheless, perovskites also face challenges, like limited exposed active sites and poor electrical conductivity. In this work, we tackled those problems by the strategic assembly of La0.8Sr0.2CoO3 nanoparticles into supraparticles as a novel class of electrocatalysts for the enhanced oxygen evolution reaction.

Electrochemical results obtained from the electrochemical beaker cell revealed that supraparticles exhibit lower overpotential at 10 mA cm-2 of 1.55 V vs. RHE compared to the nanoparticles at 1.57 V vs. RHE, along with better stability over time at 100 mA cm-2. The performance was attributed to significantly higher surface roughness, porous structures in the meso- and macroporous range, and lower resistance within the supraparticles electrode. Furthermore, post-mortem analysis using XPS after electrochemical testing of supraparticles revealed a significant increase in oxygen vacancies compared to the nanoparticles, which is linked to a partial reduction of Co3+ to Co2+. These oxygen vacancies play a crucial role in enhancing catalytic activity.

In summary, with supraparticles, we discovered a novel class of highly promising electrocatalysts because of their architecture that significantly enhances catalytic performance by ensuring a constant supply of accessible active sites, improved charge transfer, and promoted bubble management. These findings provide a strong foundation for the future development of highly effective electrocatalysts and achieving carbon neutrality.

Content

Amphiphilic Nanogels as Versatile Stabilizers for Pickering Emulsions

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Abstract

Pickering emulsions (PEs)—emulsions stabilized by nanoparticles—exhibit pronounced stability, and nanogels are increasingly investigated as soft stabilizers in PEs. Unlike PEs with solid nanoparticles, where emulsion types are defined by the contact angles of particles at the interfaces, soft nanogels swell in solvents and deform at interfaces. Up to now, there is still no clear relationship between emulsion types and nanogel properties, attributed to synthetic difficulties in accurately controlling the network hydrophobicity and insufficient understanding of the phase inversion behavior. In particular, the preparation of water-in-oil (W/O) emulsions, crucial for various applications, remains challenging.
Herein, we have developed a library of amphiphilic nanogels (NGs) with accurately tuned network hydrophobicity while keeping similar colloidal features. Our synthetic approach is based on network functionalization of reactive ester-based precursors with various ratios of hydrophilic moieties and hydrophobic (dodecyl amine - DODA) moieties. We find that W/O emulsions with these NGs are preferentially formed with increasing NG hydrophobicity, oil polarity, oil/water ratio, and temperature. Intriguingly, the phase inversion behavior can be rationalized by the differences in the Flory-Huggins parameters of NGs with water and oil ($\chi_{water} - \chi_{oil}$). Carefully balancing these parameters allows the formation of W/O emulsions from non-polar oils. Notably, emulsification at elevated temperatures enables forming metastable W/O emulsions, which remain stable after cooling to room temperature. Overall, we envision this strategy to serve as a platform for accurately tuning the properties of PEs to advanced applications such as interfacial catalysis, cosmetics, and drug delivery.

Content

Synthesis of highly-dispersed hollow silica nanoparticles and their application to composite materials

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Abstract

Hollow silica nanoparticles (HSNPs) have a core-shell structure consisting of a void core and a silica shell. Due to their unique structure, HSNPs have various characteristics, such as low thermal conductivity, low dielectric constant, and high specific surface area. By using HSNPs as fillers in composite materials, the properties can be imparted or improved to the matrix. If composite materials made of hollow particles and resin could be used as paint for building window glass, they could significantly improve the efficiency of air conditioning and save energy. However, to maximize the functionality of HSNPs, it is necessary to prepare a homogeneously dispersed composite.

In the template hollow particle synthesis method, template particles for forming the hollow core are dispersed, the dispersed particles are coated with silica for the shell, and then the template particles are removed. When emulsion droplets are applied to template particles, the core template components can be removed simply by washing with water. Furthermore, by encapsulating a dispersant within the droplets, it is possible to synthesize highly dispersible hollow particles. In this study, HSNPs were synthesized using a polyacrylic acid-ammonium solution emulsion system; composite membranes were prepared with cellulose nanofibers, and the thermal insulation properties of the composite materials were evaluated.

Structure Formation in Binary, Self-Assembling Particles by Diffusiophoresis

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Abstract

Structure Formation in Binary, Self-Assembling Particles by Diffusiophoresis
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The structure formation in evaporating droplets containing binary particle dispersions is influenced by solvent evaporation speed and particle size ratio. Drying such dispersions can yield composite or core-shell structures depending on primary particle sizes. This arrangement impacts supraparticle properties, enabling applications such as diffusion-controlled release of two active pharmaceutical ingredients. During drying, convection transports particles to the droplet interface while a concentration gradient forms in the center due to reduced particle density. This gradient generates osmotic pressure directed towards the center, creating a radial force proportional to particle size. Consequently, small particles accumulate at the supraparticle interface, while larger ones form the core — a phenomenon known as diffusiophoresis. Here, we investigate the segregation of binary particles in the formation of supraparticles as a function of size and number ratio.
Acknowledgements:
This Work is funded by the Deutsche Forschungsgemeinschaft (DFG, German Research Foundation)– Project-ID 416229255 – SFB 1411

Content

Advanced Textural and Wettability Characterization by Combining Adsorption and Liquid Intrusion with NMR Relaxometry

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Abstract

Textural properties and the wettability of nanoporous materials play an important role for their applications, e.g., in chromatography and catalysis, since they can enhance the affinity and selectivity of certain compounds. A detailed investigation of the relation between structural properties, wettability and the resulting process performance is required. Within this context, we have recently shown that NMR relaxometry can be developed into a methodology for surface area assessment of nanoporous materials immersed in a liquid phase. We also demonstrated that NMR relaxometry shows potential for fast determination of pore entrance sizes.

The quantification of the wettability inside of pores is challenging and usually requires a combination of different experimental tools. Within this context, we demonstrate that NMR relaxometry is a valuable complementary tool for the fast wettability characterization. For that, the ratio of the spin-lattice to spin-spin relaxation time (T1/T2) has been suggested in the past. However, this ratio can be affected by the wettability as well as textural properties. To address this limitation, this study suggests a novel methodology for wettability quantification based on the spin-lattice to spin-spin relaxation time ratio of the adsorbed liquid film on the pore surface. This novel approach allows for the determination of a characteristic value, solely affected by the wettability. This is demonstrated using mesoporous stationary phase materials functionalized with different hydrophilic and hydrophobic surface functional groups. The study shows, for the first time, the correlation between the T1,ads.film/T2,ads.film-ratio of water and the contact angle of the adsorbed water film determined with water vapor adsorption and water intrusion.

Content

Model-based optimization of process parameters in high energy impact additive manufacturing processes

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Abstract

Cold gas spraying is an emerging technology in additive manufacturing, known for its versatility and broad range of applications. This process enables the deposition of various materials, such as metals, ceramics and polymers, onto substrates by accelerating particles to high velocities within a Laval nozzle. In this study, a process concept was developed that enables the targeted use of cold spraying for autonomous 3D additive manufacturing. The choice of components and the design focused in particular on achieving low response times to attain the specified process parameters. To achieve optimal manufacturing quality at low cost, continuous and precise adjustment of process parameters is essential. However, due to the complex behaviour of the gas dynamics, assessing quality during manufacturing is challenging. To address this issue, two data-driven modelling approaches that connect process parameters to particle descriptors within the spray jet are described and compared: a fast and easy-to-implement radial basis function network method and a low-parametric copula-based method in order to probabilistically model the high-dimensional dependencies among particle descriptors. These two modelling approaches are illustrated through an example of data-base optimization of process parameters for a cold gas spray in free jet, but are directly applicable to non-free jet data, if available. Furthermore, both methods are extended to include 3D image data of the manufactured product in order to close the gap between free jet measurements and actual particle colliding with the substrate. In this way, 3D morphological descriptors of the product are correlated with free jet particle descriptors to further characterize and quantify the quality of the spraying process.

Content

Introducing a Novel Autonomous Process Chain for Selective Precipitation of CaCO3 and MgCO3 using Extracted Mine Tailings

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[2] Karlsruhe Institute of Technology;
[3] Rheinland-Pfälzische Technische Universität Kaiserslautern-Landau

Abstract

As the atmospheric CO2 concentration continues to rise, innovative carbon capture and storage (CCS) approaches are crucial. Carbon mineralization offers a sustainable solution by permanently capturing CO2, as Calcium and Magnesium carbonates have wide industrial applications. The abundant availability of metals (Ca, Mg) in industrial waste streams, mine tailings, and seawater enhances the viability of this approach. Mine waste utilization for CO2 capture can yield high-purity carbonates (CaCO3 and MgCO3), but fluctuating feed material composition poses challenges for product purity, particle size, and process efficiency. To address these challenges, we propose implementing and validating an autonomous, self-learning process chain for the selective carbonate precipitation. A control algorithm was designed to dynamically adapt to fluctuations in raw material composition and particle size distribution, optimizing productivity and ensuring consistent product purity. A semi-batch process model has been developed for the stepwise precipitation of high-purity CaCO3 and, subsequently, pure MgCO3. A coupled 2D population balance model for the selective precipitation process is also created as a prerequisite for the controller design and implementation. The experimental validation of the control model, a crucial part of our research, is achieved by implementing the pH-swing process. The extraction of the ions from the tailings is realized at acidic (low) pH conditions, while the precipitation of the carbonates is favored at basic (high) pH conditions. This necessitates the need for the pH swing in the solution. The influence of pH, PCO2, and seed particles are measured using continuous (PSD) and offline (IC, XRD) analytics, ensuring the validity of our results.

Content

Control and Optimization of Spray-Drying Parameters for Alumina: Influence of Spray Rate on Particle Descriptors

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Abstract

This talk addresses the impact of varying spray rates on size and shape descriptors of nano-alumina particles [1] generated through spray drying with a Büchi B-290 system, using a water-based suspension containing PVP-30 binder. The synthesized particles were examined to characterize their size distribution, morphology, and porosity, based on both low-resolution 2D inline measurements and high-resolution offline methods such as Hg-porosimetry and 3D CT imaging. The 2D inline measurements focused on determining rough particle shape and morphology in real-time, while offline analyses provided detailed descriptors for the 3D structure and porosity. Notably, variations in the spray rate led to observable shifts in particle size distributions, with shapes ranging from irregular to nearly spherical. A key objective of this research is to establish a robust correlation between the multidimensional particle descriptors derived from low-resolution 2D inline measurements and those acquired offline. This can enable a rapid and reliable prediction of multidimensional particle descriptors, facilitating real-time process adjustments without waiting for time-consuming laboratory evaluations and process shutdown. The reliable prediction of optimal process parameters (such as spray rate) corresponding to desirable particle descriptors can streamline production, reduce material waste, and ensure high-quality spray-dried products.
[1] Cottrino, S., et al. "Spray-drying of highly concentrated nano alumina dispersions." Powder technology 237 (2013): 586-593.

Real-time control of nanoparticle synthesis by spark ablation

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Universität Duisburg-Essen

Abstract

While particle processes are still largely controlled empirically at both industrial and laboratory scales, this work explores the feasibility of autonomous control for particle synthesis. Spark ablation is an electrical discharge method for producing nanoparticles from solid electrodes through evaporation and consequent homogeneous nucleation in an inert gas atmosphere potentially producing pure metallic nanoparticles, with constituent particles as small as a few nanometers. However, the resulting electrode erosion, turbulence, and inhomogeneities cause the process to be inherently dynamic and unstable. A real-time control system solves these issues by guiding the process using meaningful particle properties. The key performance indicators (KPIs), defining the output properties of spark ablation synthesis, are measured on-line, and provide insights into the mesostructure of the particles. This requires simultaneous assessment of particle mobility and aerodynamic size. The control system employs machine learning methods trained on experimental data to ensure adaptability, robustness, and precision. This data includes input parameters such as charging current, discharge voltage, and gas flowrate, as well as output KPIs like primary particle diameter, number of primary particles per aggregate, and production rate. At its core, the system features a control theory informed machine learning-based model leveraging control-theoretic pre-knowledge to enhance system stability, improve predictive accuracy and mitigate the effects of sensor noise. A key system component is a monodisperse particle formation model predicting the evolution of aggregate surface area and volume, incorporating their fractal-like nature.

Content

Continuous Aqueous Two-Phase Flotation (ATPF): Adaptive Optimal Control for Autonomous Enzyme Separation

Kim Carina Lohfink[1]; Katrin Baumgärtner; Prof. Dr. Moritz Diehl; Prof. Dr.-Ing. Hermann Nirschl
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Abstract

Aqueous two-phase flotation (ATPF) is a novel separation technique for the isolation of industrial enzymes such as phospholipase from crude biosuspensions. It combines several process steps of a typical downstream process into a single unit operation, reducing energy and costs consumption. The continuous operation mode of ATPF allows high throughputs while maintaining a high separation efficiency. For efficient separation and improved product quality, an adaptive optimal control strategy is crucial.
This work uses a laboratory ATPF plant for the development of a suitable control strategy. The basis of ATPF is an aqueous two-phase system (ATPS). The biosuspension containing the enzymes to be separated is mixed with the phase forming components of the high-density bottom phase. Three gassing units introduce gas bubbles via porous media at the bottom of the flotation tank. The enzymes accumulate with their hydrophobic areas on the bubble surfaces and are thus selectively transported into the top phase.
Online measurement technology is integrated into the plant in order to monitor various process parameters. A spatially resolved determination of the phase mixing caused by rising bubbles is realized with three electrical conductivity probes. Online UV/Vis spectroscopy is used to measure the enzyme concentration at the top phase outlet in order to determine the separation efficiency in real-time. ATPF experiments with varying process parameters are performed to characterize the process behavior. The experimental results allow the identification of a system model as the basis for the development of a suitable closed-loop control system. The autonomous ATPF process is able to respond to feed fluctuations in real time and ensures a consistently high separation efficiency.

Content

Towards Autonomous Control of Slug Flow Crystallization

Maximilian Rainer Kattner; Collin Richard Johnson; Dario Pelle; Stijn De Vries; Prof. Dr.-Ing. Sergio Lucia; Dr.-Ing. Kerstin Wohlgemuth
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Abstract

To achieve crystals of high and consistent quality, continuous crystallizers offer distinct advantages over established batch crystallizers, as they can help overcome batch-to-batch variability while allowing for improved space-time yields and product qualities such as particle size distributions (PSDs). A promising concept for ensuring these quality demands is the Slug Flow Crystallizer (SFC), a tubular crystallizer characterized by a segmented three-phase flow in which the process medium – mother liquor and crystals – is conveyed through a tempered tube, the growth zone of the crystallizer. Flow segmentation is achieved by introducing a gaseous phase via a T-mixer, creating liquid slugs in which crystallization occurs.

Due to the flow segmentation, internal Taylor vortices are induced within the slugs enhancing mixing in the liquid phase and ensuring the suspension of particles, thus reducing the risk of crystal attrition and agglomeration. In addition, the flow segmentation ensures a plug-flow-like profile under laminar conditions, preventing backmixing and allowing for a narrow residence time distribution of both the solid and liquid phase. However, while the SFC offers numerous advantages, its main challenge is the lack in long-term operability due to fouling issues.

In this contribution, we present the application of autonomous process control to the SFC. As part of process control, we present a strategy for mitigating the effects of crystallizer fouling as well as the application of model predictive control schemes with the aim of controlling the PSD of the product.

Acknowledgement: Our research receives funding by the German Research Foundation (Deutsche Forschungsgemeinschaft – DFG) in the framework of the Priority Program SPP 2364 – Project No. 504676854.

Content

Towards an autonomous powder compaction process

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Abstract

Powder compaction performed on a rotary tablet press is a dry granulation method to transfer powder materials consisting of several components into compacts (tablets). The product quality attributes are maintained during processing by adaptation of the subsequent unit operations feeding, blending and filling, which are executed continuously, and compression and ejection, which are performed semi-continuously. The interconnection of these five process steps leads to a complex control task (see Figure 1). In this respect, the aim of this study is to develop an autonomous process control system, which is to replace the human intervention into the process course to enhance the process efficiency, while guaranteeing predefined quality marks of the compact production.
In this framework the development of a sensor system for the in-situ data acquisition to enable real-time monitoring of the compaction process is required. Therefore, machine data are combined with spectroscopic information determined by a novel UV/Vis fiber optic probe incorporated into the rotary tablet press and complemented by highly specific ex-situ methods.
Then nonlinear autoregressive models with exogenous inputs (NARX), which account for the process inherent disturbances, are developed for the process control based on the in-situ data. Model performance is evaluated using systematically designed experiments and validated in real-time scenarios.

Content

Real-time prediction of aggregate structure based on bivariate aerosol dynamics

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Abstract

Precise control over nanoparticle synthesis in gas-phase processes remains a significant challenge due to the complex, nonlinear dynamics of particle formation in systems dominated by coagulation and sintering. This work presents a computational framework combining a Monte Carlo (MC) method and a convolutional neural network (CNN)-based surrogate model to accelerate predictions of bivariate particle descriptor distributions. The MC method predicts the evolution of particle surface area and volume distributions over time at isothermal conditions, capturing the fractal-like nature of aggregates and their effects on formation dynamics. Monomers are assumed to be instantaneously released, and nucleation theory is negligible due to the high degrees of supersaturation in synthesis processes such as spark ablation. Validation against established 1D and 2D models demonstrates high agreement, emphasizing its accuracy in capturing particle formation dynamics. The CNN surrogate model leverages 2D histograms to predict time-dependent distributions for variable temperatures, achieving a 15.000-fold reduction in computation time compared to the MC method and thus reaching real-time capability, while maintaining sufficient accuracy. Additionally, the model's differentiable nature enables optimization of temperature profiles. Thus, this work demonstrates the potential of integrating advanced MC methods with neural networks to balance computational efficiency and predictive accuracy.

Content

Autonomous structure formation in fluidized bed spray agglomeration

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Abstract

Spray agglomeration is fluidized beds is a common technology for the formulation of structure bulk materials, for example in fine chemicals and foods. In this process, a liquid-based binding agent (e.g., suspension or solution) is sprayed on a fluidized bulk material. The sprayed material deposits on individual particles. New structures – agglomerates – form, if several sprayed particles collide and solid bridges are formed. The process behavior, the agglomerate structure and functionality depend on the agglomerates' structure, e.g., the number of neighboring particles (agglomerate strength) or fractal dimension as a measure of accessible surface area (e.g., for later dissolution of the aggregated material in liquids).
In this contribution, we will present recent results on the autonomous structure formation in fluidized bed spray agglomeration. We will report on methods and strategies to obtain favorable agglomerate structures by controlled manipulation of the operation conditions. Aspects comprise the systematic offline characterization of agglomerate structures to establish process-structure relationships, development of online methods to assess structure formation in situ, and the development and evaluation of feedback control approaches to obtain desired agglomerate structures.

Content

Towards Autonomous Nanoparticle Synthesis: Model-based Control and Al-Doped ZnO Crystallization

Marcel Kévin Jiokeng Dongmo[1]; Guohui Yang[2];
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[2] Karlsruhe Institute of Technology, Institute of Mechanical Process Engineering and Mechanics, Process Machines Group

Abstract

This project develops a model-based control framework for synthesizing aluminum-doped zinc oxide (AZO) nanocrystals with tailored properties, such as size distribution, aimed at enhancing applications like solar cells. Using the sol-gel method, control over particle morphology is achieved by optimizing temperature. A soft-sensor-based predictive control strategy is designed to manage the crystallization process. The system integrates a process controller, a model-based soft sensor, and online UV-Vis method. This enables monitoring of AZO mass concentration and real-time estimation of system states and particle properties using a moving horizon estimator (MHE) combined with model predictive control (MPC). The synthesis is modeled by a system of differential equations, with parameters calibrated via SAXS and DLS measurements. This model facilitates the reconstruction of the AZO growth. The key contribution is the controlled nanocrystallization to enable autonomous synthesis. Highlights include a population balance model, online UV-Vis spectroscopy, and a linear approximation of the nonlinear system. This approximation is achieved via the dynamic mode decomposition with control (DMDc), resulting in a fast-computing time-discrete linear system. The resulting hybrid system forms the foundation of the MHE-based MPC framework, which minimizes differences between measured and estimated states (for MHE) and desired and estimated particle properties (for MPC), enabling state estimation and real-time control. The methodology is applied to AZO synthesis, reconstructing size distribution based on estimated concentrations, demonstrating property control. This work achieves particle property control during synthesis, broadening the scope of applications for tailored nanomaterials.

Content

Prilling of polymers – Investigation of viscoelastic jet breakup

Vasileios Champilomatis[1]; Yavuz Emre Kamis PhD; Toon Nieboer
[1] Kreber

Abstract

Liquid jet breakup and droplet formation are phenomena closely related to prilling, a particle technology in which a solid material is turned into spherical particles from its molten state through jet breakup. Prilling experiences challenges when it comes to processing polymers, as a result of the viscoelastic behavior shown by jets resulting from the dispensing of molten polymers. Our research focuses on predicting viscoelastic liquid jet breakup based on their material properties and flow parameters, aiming to investigate the feasibility and limits of polymer prilling.

In this study we investigate how jet breakup lengths are affected by different Oh, De and We numbers by performing lab experiments using PEG. We have developed a setup to control and monitor jetting, droplet formation and solidification behavior of molten PEG using dynamic image analysis. With the developed setup we can induce jet breakup, track the breakup length and size distribution of the resulting droplets, and collect solidified particles. This study has proven the feasibility of prilling as finishing technology for PEG, by producing prills of D50 equal to 1.7mm in the lab. The insights have been used to connect material and flow parameters to predict and model the resulting jet breakup lengths of polymer melts and determine the limits of the technology based on the viscoelasticity of the molten materials.

The ongoing research focuses on translating the results to different polymers, in particular PVA, PLA and HPMC by studying the effect of the prilling process parameters, such as cooling rate and melt flow rate, at pilot scale. The goal is to establish prilling as a competitive polymer finishing technology, aiming to produce prills of D50 equal to 1.7mm and sphericity higher than 0.8.

Content

Prilling slurries – Determining the influence of particle size and solid content on jet breakup and droplet formation.

Dr. Kilian Schnoor; Yavuz Emre Kamis PhD; Toon Nieboer
Kreber

Abstract

Prilling of slurries has gained significant interest in the past years as a production method for N,P and K fertilizers.There is good understanding of slurries but only limited research is being done to investigate their jetting,jet breakup,droplet formation and solidification behaviour.We develop a model for the prediction of material behaviour based on the PSD of the added solids and solid content in the melt.We aim to estimate material properties based on the observed material behaviour.

We investigated the behaviour of slurries with particles below 300µm solid contents up to 40w/w-%.We prepared a model liquid with different particles and concentrations.We studied the flow behaviour of these suspensions under different shear rates and their sedimentation characteristics.The results validate:larger particles sediment quicker and increase viscosity,smaller particles lead to a shear-thinning behaviour.

We model the jet formation,jet breakup and droplet formation and validate it using our jetting setup and the integrated DIA by capturing the jet breakup,droplet formation and focus on the jet breakup length, the droplet size and shape,and the periodicity of the jetting process. We refine and adapt the model to allow modelling and prediction of the whole prilling process using physical material properties.

The results show that particle size and solid content influence the non-Newtonian behaviour of the slurries.Smaller incremental steps will further develop the model. In the future the behaviour of materials can be used to estimate the non-Newtonian properties of unknown materials,allowing the characterization of materials where the determination of the non-Newtonian properties has proven extraordinarily difficult due to high melting points or high vapor pressure.

Content

Investigating the Role of Long-Range Interaction Forces on Segregation in Drying Droplets via CFD-DEM Coupling

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Abstract

Porous particle aggregates offer significant potential for diverse applications such as catalysis and pharmaceuticals. Their functionality is largely determined by their microstructure. Consequently, understanding the mechanisms underlying the formation of these structures is essential for effectively controlling their microstructure during fabrication. One possible production route of such supraparticles is spray drying. By carefully selecting the process and formulation conditions, the outcome can be effectively controlled. For instance, mixtures of small and large particles are known to form segregated aggregate structures, with smaller particles mainly residing on the surface, encapsulating larger particles concentrated near the particle centre. The segregation strength is mainly influenced by factors such as particle size ratio and particle concentration. However, recent studies have suggested, that interparticulate forces such as electrostatic repulsion and van der Waals forces might additionally influence the degree of segregation. These findings are further investigated in this study using simulations of single suspension droplets. To achieve this, coupling of computational fluid dynamics (CFD) with discrete element method (DEM) is employed, using the volume of fluid method (VOF) to take into account the gas-liquid interface. VOF-DEM coupling is realized by a capillary force model, while interparticulate forces are considered via classical contact models and DLVO forces. The influence of the different parameters of the DLVO model on the structure formation is examined and explained with established theories about “small on top” stratification in drying films. Based on this knowledge, guidelines for the targeted design of hierarchical supraparticles are proposed.

Content

Molten metal droplets as a versatile tool for alloy development

Dr.-Ing. Nils Ellendt
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Abstract

The design of metallic alloys typically revolves around specific applications and their required mechanical or functional properties. With the advent of advanced synthesis processes like additive manufacturing, alloy design must also account for fluid properties to enhance processability during atomization and manufacturing. This study demonstrates a novel approach using single molten metal droplets to evaluate a broad range of properties in a single experiment via a pneumatic high-temperature droplet generator.
As droplets form and detach from a liquid, they undergo initial deformation, inducing oscillations recorded using high-speed imaging. Quantitative image analysis extracts the oscillation frequency, linked to surface tension via the Rayleigh equation, and the decay of oscillation amplitude, related to melt viscosity through the Lamb equation. By varying the melt temperature, temperature-dependent properties, including surface tension, can be measured. The temperature dependence of surface tension drives Marangoni convection in additive manufacturing melt pools and is one key parameter for the process.
During free fall in an inert atmosphere, the droplet cools through convection and radiation, with solidification rates governed by heat dissipation. This enables microstructural screening via droplet size variation. Additionally, when droplets impact a cold copper plate, much higher cooling rates are achieved through directed heat flow, creating distinct solidification conditions. This dual approach facilitates microstructural screening and property determination within the same experimental setup.

Content

Effects of extrusion mixing on powder properties and processability of LFP mixtures

Milena Lux
Fraunhofer IWS

Abstract

Dry coating of battery materials has emerged as a promising alternative to state-of-the-art slurry coat-ing, offering advantages in terms of cost-effectiveness and sustainability. Within this work, correla-tions between the process steps of dry mixing and calendering for the dry coating of lithium-iron-phosphate (LFP) cathodes were investigated.
Therefore, active material, conductive additive and PTFE binder were mixed in a continuous twin-screw extrusion process at different intensities via variation of the screw configuration, e.g. the kneading section. In the kneading zone the premixed materials were heated in order to facilitate fibrillation by mechanical stressing with different kneading elements.
Subsequently the granules were crushed down to smaller sizes in a shaping zone at lower temperatures.
Suitable characterization methods for the dry mixed powders were applied to conclude on process-property relationships between mixing intensity and granule properties. Intensive mixing with a high specific mechanical energy input results in a more homogeneous and fine PTFE fibril network smaller granules and more compact granule shapes.
These differences in micro- and macrostructure lead to a higher electrical resistivity of the com-pressed granules due du a stronger fibrillation. Bulk and tapped density as well as flowability of the granules are influenced by the macrostructural particle shape and increase after higher mechanical stressing due to the less rough granules surfaces and higher sphericity.
The processability of extrusion-mixed granules in the calender gap highly depends on their specific mixing histories. In the patented DRYtraec® process the film formation takes place in a single step. The calender rolls rotate at different speed inducing tangential shearing forces on the granules. The film then adheres to the faster rotating roller and can be laminated onto a substrate foil subsequently. The value of this shearing ratio highly influences film thickness and areal mass. Low shearing ratios may lead to thick and brittle layers or to exceeding the

Content

maximum torque of the calender motor while high shearing ratios result in thin freestanding films that do not adhere to the faster roller. These two domains have to be avoided in order to ensure processability of mixed products in the DRYtraec process.
The processing window for the extruded granules in this study broadened for high mixing intensity compared to softer kneading, resulting in thinner electrodes due to the higher sensitivity of a fine PTFE fibril network to shearing forces within the calender gap. A lower number of fibrils in the more softly kneaded granules result in less contact points between PTFE and the calender rolls which increases the domain of freestanding films.
Characteristic properties of the electrodes were described via adhesion and conductivity measure-ments. As already observed for the granules, strong mechanical stressing in the extruder leads to a higher electrical resistance and to stronger adhesion. In electrochemical tests the electrodes made from strongly kneaded granules demonstrate a slightly inferior cell performance at higher C-rates.
These results highlight the importance of understanding correlations between dry coating process steps and indicate the possibility to tailor the extrusion mixing process towards suitable powder properties for dry calendering as well as optimized electrode characteristics.

From Pores to Performance: Multi-Scale Analysis of Catalyst Layers for PEM Fuel Cells

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Abstract

Proton Exchange Membrane Fuel Cells (PEMFCs) are key to sustainable energy solutions, but high costs, limited stability, and sluggish oxygen reduction reaction (ORR) kinetics hinder their adoption. The catalyst layer's pore network and morphology are critical for improving mass transport, catalyst site exposure, performance, and durability.
Catalyst layer microstructure is influenced by ink formulation and fabrication parameters. Traditional characterization methods—such as mercury intrusion porosimetry (MIP), nitrogen adsorption, and microscopic imaging—offer valuable insights but are limited in capturing features across scales from nanometers to micrometers. To address this, we developed a multi-scale structural analysis method combining MIP, microscopic imaging, and digital processing of FIB-SEM cross-sections for both qualitative and quantitative assessments of catalyst layers from 10 nm to 10 µm. Applying this method to investigate hot-pressing effects during catalyst-coated membrane (CCM) fabrication revealed distinct microstructural changes based on ink recipes and processing conditions. Higher hot-press pressures typically reduced porosity and compacted layers, improving internal resistance and adhesion. However, some cases showed increased secondary porosity at higher pressures, highlighting the influence of layer composition on pore formation.
Our methodology offers a comprehensive framework for optimizing PEMFC catalyst layers and is being expanded to battery and anion exchange membrane (AEM) electrolyzer electrodes. By linking microstructure to performance, it supports the development of efficient, durable, and scalable energy storage technologies.

Content

Proof of physical work for a more sustainable decentralized consensus in blockchain technology

Dr.-Ing. Frank Rhein
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Abstract

Cryptography and blockchain technology play a pivotal role in shaping a trustworthy and transparent future. Digital one-way functions, exemplified by cryptographic hash functions like SHA-256, form the backbone of secure communications and decentralized ledgers. They readily provide a deterministic output for any given input, while it is impossible to analytically find the corresponding input for any given output. The most prominent application example is the cryptocurrency Bitcoin. Here, the integrity of the blockchain is maintained by the so-called proof of work, where network participants (miners) compete to find solutions for specific inverse problems. This can only be achieved by trial and error and requires computational power (work) on a massive scale.

The physical world is inherently complex, with phenomena that cannot be fully described by closed analytical expressions, like e.g. interactions of heterogeneous particle structures with light. This motivates the search for a novel physical one-way function that fulfills the same criteria. Based on p-OWFs, a proof of physical work can be conceptualized that promises a significant CO2 reduction for decentralized blockchain applications.

This talk presents the results of a recently published study. Boundary conditions for the security requirements were established and discussed as well as experimentally investigated for a specific setup based on printing and optical analysis of pigment particles. This setup promised to be mathematically unclonable, steady, reproducible, collision resistant and non-invertible and illustrates the feasibility of a physical one-way function. Based on this, a framework for a proof of physical work was conceptualized. This work initiates a progressive, interdisciplinary field of research.

Content

Microplastic pollution in aquatic environments: A meta-analysis of influencing factors and methodological recommendations

Linus Hartz; Lisa Grabinski; Prof. Dr.-Ing. Samir Salameh
FH Münster

Abstract

Microplastics (MPs), which are defined as plastic particles smaller than 5 mm, have emerged as pervasive pollutants in marine and freshwater ecosystems. They raise increasing ecological and public health concerns. These pollutants originate from various sources, including primary microplastics, which are intentionally manufactured for commercial applications, and secondary microplastics, which result from the fragmentation of larger plastic debris due to environmental degradation processes. Despite rising awareness and an increasing number of field studies, it remains challenging to make comparisons across studies due to inconsistent sampling methodologies and reporting metrics.
This study presents a meta-analysis integrating and analyzing microplastic concentration data from over 60 studies in European freshwater and marine environments. Our goal is to better understand the interplay between anthropogenic and methodological factors that influence microplastic pollution levels, as well as to formulate recommendations for improving methodological harmonization in future research.
We consider a range of potentially influential parameters in our analysis, including sampling latitude, proximity to wastewater treatment plants (WWTPs), population density, the mesh size used during sampling, distance to coastlines, seasonal variations, and wind speeds at the time of sampling. Using a combination of statistical correlation analysis (Pearson's r, Kruskal–Wallis test, and Mann–Whitney U test) and geographic contextualization, we evaluate the relative impact of each parameter on measured microplastic (MP) concentrations.
Key findings from the meta-analysis reveal that mesh size significantly affects concentration results in marine environments, where smaller mesh sizes consistently yield higher microplastic (MP) counts. However, this correlation was not observed in freshwater datasets, which suggests that primary microplastics dominate rivers and lakes, likely due to shorter

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residence times that preclude significant fragmentation. These results challenge the common assumption that proximity to WWTPs is the main driver of microplastic pollution. While WWTPs remain a known point source, our analysis indicates that other factors, such as surface runoff, atmospheric deposition, and diffuse urban or industrial discharges, play a more substantial role.

Interestingly, population density within a 25–50 km radius around sampling locations correlates moderately with MP concentration levels, particularly in freshwater systems. This suggests that localized anthropogenic pressures are key contributors to MP inputs. By contrast, neither distance to coastline nor wind speed showed statistically significant relationships with MP concentration, indicating that existing correction factors for wind-induced mixing may be overapplied or context-dependent.

Seasonal influences are complex, yet they exhibit distinct patterns when categorized by climatic region. For instance, higher concentrations in Mediterranean summer samples likely relate to tourism. Meanwhile, Northern European spring peaks coincide with snowmelt and runoff events. These findings highlight the importance of considering seasonal variations in the context of regional hydroclimatic and anthropogenic activity patterns.

Beyond correlational insights, this work critically evaluates the impact of the sampling methodology itself. Variability in mesh sizes, sampling depths, and volumes across studies limits comparability and introduces bias, particularly in particle size distribution. Based on the distribution of methods among the reviewed studies and the statistical robustness of the results, we propose a set of harmonized best-practice recommendations. These recommendations emphasize using volume-reduced Manta trawls with a mesh size of 300–350 µm, performing replicate sampling, and using flowmeters to quantify volume.

In conclusion, our study emphasizes the multifactorial nature of microplastic pollution and the need for standardized sampling and data reporting approaches. By consolidating evidence across studies and highlighting methodological pitfalls, this meta-analysis enables more reliable comparisons and paves the way for a harmonized data landscape in microplastic research. Future efforts should extend this analysis beyond Europe to validate global patterns and explore the effects of post-sampling processing techniques, which this study did not fully address.

Synthesis of Iron-Oxide Supraparticles for Enhanced Antibacterial and Photothermal Therapy

Dr. Mohaned Hammad[1]; Adil Amin[1]; Dr. Cristian Ursu[2]; Dr. Irina Rosca[3]; Dr. Valentin Nica[4]; Prof. Dr. Hartmut Wiggers[5]; Prof. Dr.-Ing. Doris Segets[1]

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Abstract

Antimicrobial resistance is a growing global health threat, with drug-resistant bacteria like Escherichia coli and Staphylococcus aureus causing infections that are difficult to treat with conventional antibiotics. The rapid emergence of antibiotic-resistant bacteria highlights the urgent need for novel therapeutic strategies. Photothermal therapy, using nanoparticles like iron oxide to convert near-infrared light into heat, offers an effective approach for bacterial eradication. However, challenges such as nanoparticle agglomeration and instability limit their effectiveness.

Herein, we present a scalable spray-drying method to synthesize iron-oxide supraparticles designed to enhance antibacterial performance. These supraparticles exhibit larger size, rough surface morphology, and high porosity, improving functional properties. Compared to their nanoparticles counterparts, iron-oxide supraparticles show superior peroxidase-like activity, efficiently generating hydroxyl radicals to disrupt bacterial membranes, denature proteins, and damage DNA. The antibacterial effectiveness of the iron-oxide supraparticles was tested against Escherichia coli and Staphylococcus aureus under near-infrared irradiation. They significantly outperformed nanoparticles in bacterial cell death. Their high porosity and rough surface enhance bacterial adhesion, while their stability in biological environments prevents agglomeration and ensures sustained activity. Importantly, these supraparticles demonstrated minimal cytotoxicity, maintaining high viability in human dermal fibroblast cells.

This study demonstrates the potential of iron-oxide supraparticles for antimicrobial therapies, optimizing photothermal properties to overcome bacterial resistance while minimizing host cell toxicity.

Content

AI-Enhanced particle model generation: Generative Adversarial Networks and Diffusion Transformers in particle technology

Konstantinos Giannis; Jiqian Guo PhD; Somayeh Hosseinhashemi; Christoph Thon; Prof. Dr.-Ing. Carsten Schilde

Abstract

As particle technology continues to advance, traditional methods for 3D particle reconstruction, such as X-ray Computed Tomography (XRCT), face challenges due to their resource-intensive and repetitive nature. Dynamic image analysis tools like QicPic and Camsizer offer faster alternatives but are limited by the small number of 2D images generated. To overcome these limitations, we introduce two innovative, AI-driven approaches—Generative Adversarial Networks (GANs) and Diffusion Transformers (DiT)—that enable the efficient reconstruction of high-fidelity 3D particle shapes from 2D images.The GAN-based methodology generates 3D models starting from Gaussian noise, projecting them into three orthogonal 2D views. A discriminator evaluates these projections against experimental data to refine model accuracy. A conditional GAN variant further enhances this approach, enabling the reconstruction of complex 3D shapes from a single 2D image without requiring 3D ground truth data. Complementarily, Diffusion Transformers leverage attention-based denoising to iteratively refine noisy embeddings, capturing intricate global relationships and long-range dependencies to produce precise 3D representations.Both techniques operate independently, offering flexible and scalable solutions for particle shape modeling. The 3D models generated through these methods are particularly beneficial for enhancing Discrete Element Method (DEM) simulations, providing improved accuracy for analyzing granular materials. By addressing the limitations of conventional reconstruction approaches, this framework offers a significant leap forward in particle technology, contributing to both research and industrial applications.

AI-Assisted Characterisation of Pharmaceutical Powders

**Dr. Mozhdeh Mehrabi PhD[1]; Dr. Arash Rabbani PhD[1];
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Dr. Mehrdad Pasha PhD[2]; Dr. Jasper Jammaer PhD[2];
Prof. Dr. Mojtaba Ghadiri PhD[1]**
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Abstract

In the early stages of drug product development, the limited amount of Active Pharmaceutical Ingredients (APIs) available makes it difficult to fully understand the properties of the powders toward their manufacturability. This shortage makes it difficult to select the right manufacturing platforms at these early stages.
To tackle these complex challenges, an AI-assisted tool is being developed to improve the powder characterization process by using Artificial Intelligence to model critical powder flow properties. In this study, a diverse range of pharmaceutical powders are carefully selected to ensure a comprehensive representation. This diversity renders them attractive for AI modelling, as it enables the system to learn and model the behaviour of powders with different characteristics
Current work on relating the dispersibility and flowability of such powders to the single-particle properties and process dynamics will be presented. Techniques developed in our laboratories, such as the Drop Test Method for adhesion and Ball Indentation Method for bulk cohesion are used along with other widely established characterization methods. The data are then used by AI-based tools to establish underlying correlations and tested against a separate set of data. This provides a tool for formulation development and enables rapid estimation of powder flow properties. The approach will streamline the characterization process and provide valuable insights for improving pharmaceutical manufacturing workflows.

Content

AI-Assisted Prediction of Particle Impact Deformation Simulated by Material Point Method

Saba Saifoori; Somayeh Hosseinhashemi; Mohammad Alasossi; Prof. Dr.-Ing. Carsten Schilde; Saeid Nezamabadi; Prof. Dr. Mojtaba Ghadiri PhD

Abstract

A hybrid AI framework has been adopted to link the impact deformation of elastic-perfectly plastic particles with their material properties and impact velocity. Initially, Material Point Method (MPM) is employed to simulate the impact of an elastic-perfectly plastic particle with a rigid wall, covering an extensive range of material properties and impact velocities. The simulation results are then integrated into the AI framework to establish the relationship between the input and output parameters. Consequently, dimensionless equations are derived
to predict the equivalent plastic strain and deformation extent based on the material properties
and impact velocity of the particle, showing a strong agreement with the MPM results. The identified equations reveal that both the equivalent plastic strain and deformation extent depend
on and can be determined from the yield strength of the material as well as the fraction of the incident kinetic energy that is spent on inducing plastic deformation. The validity of the equations is verified by comparing the MPM and predicted values of the equivalent plastic strain and deformation extent for cases with material properties and impact velocities beyond the initial dataset used for developing the equations. The equation identified by the framework
for prediction of the deformation extent is further validated by impact experiments. Additionally, a correlation is suggested to estimate the coefficient of restitution based on the material properties, covering a wide range.

Content

Development of a Lagrangian sensor for in-line measurements in fluidized bed spray granulation

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Abstract

Fluidized bed spray granulation is widely used in industries such as pharmaceutical, food, and chemical manufacturing to produce high-quality granular solids. Reliable monitoring and control of this process are essential to ensure product quality and prevent anomalies, such as hot spots or over-wetting. Conventional methods rely on stationary sensors and off-line measurements of samples, which often to do not provide the information necessary to identify local issues. To address this limitation, this study investigates the first steps in the development of a Lagrangian sensor capable of in-line monitoring of coating layer formation in a fluidized bed.

Electrical impedance spectroscopy (EIS) was employed to characterize the electrical properties of sodium benzoate, which is used as a model material for both the particle bed and aqueous spray solution. Drying experiments of sodium benzoate solution droplets on printed circuit boards (PCBs) with differently shaped electrodes (capacitive and conductive) were conducted. To correlate the impedance and phase angle to the layer properties, EIS was applied several times throughout the drying process. Furthermore, the sodium benzoate layer was characterized with regard to water content and dry layer thickness.

For all PCBs tested, the experimental results demonstrate, that EIS effectively correlates impedance with water content as well as layer thickness, proving its potential for in-line monitoring of coating layer formation in fluidized bed spray granulation. Furthermore, circular electrodes showed the best performance with regard to impedance range and symmetry, making them the preferred choice for future sensor particle development.

Content

Opti-CountTM Particles: Number Concentration Nanoparticle Standards with Certified Number and Size Using the Optical Single Particle Counter LUMiSpoc®

Dr. Matthew Hood[1]; Elia Wollik[2]; Dr. Susan Strohschein-Lerche[1]; Dr. Holger Woehlecke[1]; Bárbara Riera Rousset[3]; Dr. Cornelia Hunger[3]; Dr. Kyriakos Eslahian[3]; Prof. Dr. Dr. Dietmar Lerche[2]
[1] Dr. Lerche KG; [2] LUM GmbH; [3] Applied Microspheres GmbH

Abstract

Products containing nanoparticles are increasing annually, raising concerns about health and environmental risks. In response, regulations are tightening, requiring clear definitions of nanomaterials. In the EU, nanomaterials are defined as particles smaller than 100 nm that comprise at least 50% of the sample by number concentration. Other countries classify particles below 300 nm as nanomaterials due to their health impacts. Accurate characterization of number-based particle size distributions is critical to enforcing these regulations. While new methods are being developed to measure number concentrations in this size range, the lack of reference materials—used for validating these methods—remains a challenge, with no existing standards currently available.

To fill this gap, we developed a novel surfactant-free submicron reference material (OptiCount™, Dr. Lerche KG), composed of 400 nm polystyrene particles suspended in an aqueous buffer. Two concentrations, 10^6 and 10^8 particles/ml, were produced to cover ranges measurable by most devices. Particle size and number concentration were determined using the LUMiSpoc® (LUM GmbH) optical single-particle counter, which uses hydrodynamic focusing to guide particles through a laser while detecting scattered light in forward and sideward directions. Adjustable flow rates minimized coincidence events, and Mie theory was applied to convert scattering intensities into particle sizes.

Certified values were calculated and expanded combined uncertainties estimated per ISO Guide 35 and ISO 17034, yielding < 5% uncertainty for particle size and < 15% for number concentration. This work establishes a foundation for developing standards for particle counting in the nano- and submicron size ranges, meeting critical regulatory need

Content

Phase Tracking in Fluidized Beds via Electrical Capacitance Volume Tomography and Borescopic High-Speed Camera Imaging

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Abstract

Fluidized bed systems have been used in industry for around 100 years. However, for bubbling fluidized beds, obtaining 3-D data of bubble behavior with methods like positron emission particle tracking (PEPT) or magnetic resonance imaging (MRI) is complex and very costly. In the case of circulating fluidized beds (CFB), the flow behavior of single particles and clusters in the riser is still not fully understood, due to complex and fast particle movements, which are hard to track with most sensors. To improve existing processes, a better understanding of this topic is of great interest.
For this work, electrical capacitance volume tomography (ECVT) is used in a BFB for bubble detection, to evaluate bubble diameter, frequency, and rise velocity. With a novel phase detection algorithm, bubble migration toward the radial center of the bed, and bubble coalescence and splitting were detected [1].
In a CFB system ECVT is compared to capacitance probes and borescopic high-speed camera particle tracking velocimetry (PTV). The experiments focus on turbulent and fast fluidization regime of an 8 m high and 0.1 m diameter riser with Geldart group A particles. ECVT and capacitance probe measurements lead to similar solids volume fraction trends. With borescopic PTV, particle and cluster trajectories were evaluated at different radial positions. In the radial center, mostly upward particle movement was detected. Horizontal and vertical particle movement were evaluated individually. With all three methods a 2 cm wide core flow was observed in the fully developed region.
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Content

Innovative Development of Metal Sulfide Nanoparticles in the Gas-Phase

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Abstract

The development of an innovative reactive spray technology, derived from the well-established gas-phase metal oxide synthesis route, offers extensive possibilities for producing non-oxide nanoparticles. Among these, metal sulfides are particularly promising due to their potential in electrochemical and photochemical applications requiring high surface area materials. This study explores a multi-scale approach by combining flame spray processes with single-droplet combustion to strengthen the understanding of gas-phase metal sulfide formation. Many different binary, ternary, and doped metal sulfides were synthesized in an oxygen-lean, sulfur-rich environment, using single-droplet combustion of copper 2-ethylhexanoate dissolved in tetrahydrothiophene as a model system for Cu_2S. This approach provides critical insights and expertise that could enable the next generation of gas-phase technologies for scalable synthesis of functional metal sulfides. Among the synthesized materials, tin-doped In_2S_3 particles were investigated for their potential in photoelectrocatalysis. The ternary phase, $In_4Sn_3S_{12}$ (43% Sn-doped In_2S_3), demonstrated significantly enhanced photocatalytic activity compared to pure In_2S_3, attributed to improved charge transfer resistance and a reduced work function energy. These findings underscore the potential of this innovative approach in advancing the scalable production and application of high-performance metal sulfides in energy and environmental technologies.

Content

Bottom-up synthesis of core@shell catalysts for particle size effect investigations: current status and perspective

Kerstin Wein; Dr. Jens Friedland; Prof. Dr. Robert Güttel
Ulm University

Abstract

Nanoparticles (NPs) are widely applied as solid catalysts, where the activity, selectiv-ity and stability are governed by particle size and distribution.[1] Especially in wet-chemical NP approaches, however, the controllability of the size is still lacking a sys-tematic understanding of the correlation between synthesis parameters and the re-sulting sizes. Hence, we focus on understanding the impact of operation conditions during NP synthesis to tailor NP size and distribution. Therefore, reaction parame-ters (e.g., temperature, heating rate, reactor volume, surfactant) are systematically varied, while both batch and continuous NP synthesis strategies are investigated.

The NP size and distribution are compared for batch and continuous synthesis and shows the impact of surfactant molar mass. In general, the following key state-ments can be made: The NPs from batch synthesis exhibit significantly smaller par-ticle diameters than those from continuous operation. In both operating modes, the choice of surfactant is decisive. A minimum particle size at medium molar mass of surfactant is found, while a narrow distribution is obtained for higher molar masses. Furthermore, smaller particle sizes are obtained at higher temperatures. Our results provide the systematic experimental basis to understand wet-chemical NP synthesis with the aim of tailor the particle size and distributions in batch and continuous mode.

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Synthesis of Carbon-coated Aluminum Nanoparticles by Laser Ablation in Liquids

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Abstract

The synthesis of aluminum nanoparticles (nAl) via laser ablation in liquid (LAL) hydrocarbons is explored, focusing on their morphology and tunable efficacy in energetic composites. nAl is highly desired for energetic applications because of its high enthalpy and reaction rate. However, passivation is needed for nAl to avoid unwanted ignition. A naturally formed oxide layer, when exposed to air, achieves this passivation, but it is also “dead weight,” which absorbs the heat release. A carbon-coating around nAl would passivate the nanoparticle and allow further heat release in an oxidizing atmosphere. Therefore, using liquid hydrocarbons as the ablation medium, the synthesized nAl is passivated with a carbon coating, preventing rapid oxidation and enhancing its stability. Moreover, the carbon coating provides other physical properties (such as electrical conductivity), which are advantageous as a filler in a composite. Parameters such as laser fluence, spot diameter, fluid height, and the choice of fluid can be adjusted to control the carbon coating thickness and crystallinity and, in turn, the energetic properties of the particles. This coating not only reduces unwanted interfacial reactions but also contributes additional enthalpy through carbon oxidation, which is also studied. Furthermore, post-processing cold plasma treatments may be used to functionalize the surface. Characterization of particle size, composition, and morphology provides fundamental insights into the energetic potential of nAl, and the tunability of its reaction rate in energetic composites is evaluated.

Predicting powder flows in convective mixers through multi-scale rheology

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Abstract

Predicting powder flows in convective mixers through multi-scale rheology
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Powder handling often involves a dry mixing operation, whether that is a preliminary blending stage before another process, or the blending together of different powders that are going to be the end product. In the food industries, convective mixers are commonly used for powder blending processes. These mixers typically feature one or two agitation mobiles–such as ribbons, screws or paddles–within the mixing vessel. During operation, three mixing mechanisms occur: convection, shearing, and diffusion. Researchers often focus on convection, the macroscopic movement of bulk powder from one zone to another, and diffusion, which influences the fineness of the blend. However, the shearing mechanism–despite its potential impact on particle interactions and blending quality– is frequently overlooked or poorly characterized.
Therefore, the analysis of powder flow induced by an agitation device requires further investigation to optimize the design and operation of industrial mixers. Systems equipped with an agitation mobile, like convective paddle mixers, are primarily described by the torque resulting from the interaction between the paddles and the powder bed. Several studies have identified, through dimensional analysis, correlations between a torque-related parameter, the power number, and an agitation speed-related parameter, the Froude number [1], [2]. However, these correlations often neglect the characteristics of the powder being agitated.

Content

Therefore, our study aims to incorporate these characteristics into a correlation between two dimensionless numbers: the effective friction coefficient μeff and the inertial number I.
Our research involves mixers with capacities ranging from 0.3 l to 3000 l and diverse configurations, including variations in the number of paddles, the number of shafts and their orientation—horizontal and vertical. It encompasses rheological measurements and a phenomenological study of powder behavior, particularly focusing on the dense flow regime within shear bands–a mesoscopic scale–formed during agitation on either side of the paddles [3].
Experimental results validated that the flow in the agitation systems used for the study occurs indeed in the dense regime [4], [5] and that the developed μ(I)-rheology law can be transposed from one mixer to another, regardless of configuration. This rheological law links macroscopic characteristics (e.g., torque, mixer dimensions, operating conditions) to the microscopic characteristics of the powder. This approach enables the description of powder behavior across different regimes and facilitates scale-up.
Moreover, it was found that μ(I)-rheology exhibits good predictive capabilities for flow behaviors of powders with similar particle shapes but different sizes, rather better for deep powder beds than shallow ones [6].
Furthermore, the application of μ(I)-rheology to deep powder beds allows all data to collapse onto a single curve. This indicates that μ(I)-rheology could serve as a unifying tool, despite large variations in torque measurements across different fill levels and mixer configurations.
Ultimately, the adopted modeling approach enhances the understanding of powder agitation dynamics. In particular, it facilitates the identification and spatial localization of the shearing phenomenon that arises from the interaction between the agitation device and the powder bed. These insights contribute to a more comprehensive framework for analyzing and predicting powder behavior under mechanical agitation.

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Recurrence CFD Application to Study the Mixing Behavior of Particles in Fluidized Bed

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Abstract

The main challenge of the approach of coupled computational fluid dynamics (CFD) and discrete element method (DEM) method, which is frequently applied for describing gas-solid processes, arises from the high computational costs due to the short time steps required for the DEM part. Recent developments in data-driven modelling can address the high computational costs associated with long-duration simulations of fluidized beds. Recurrence CFD (rCFD) samples the global flow field and creates a database by comparing time steps pairwise based on a recurrence norm representing a flow field variable [1]. On top of this time-extrapolated primary flow field, particle specific information in propagated.
The primary goal of this study is to demonstrate the application of rCFD for describing the process behaviour of gas-solid fluidized beds over extended time durations by comparing the mixing index from experiments and rCFD simulation, while also highlighting the computational cost advantages of rCFD over the traditional approach.
In the first step a full CFD-DEM simulation of the pilot scale fluidized bed GF25 (Glatt GmbH, Germany) as shown in Figure 1(a) is established. Figure 1(c) indicates that for the rCFD simulation, the monitoring time step should be around $\Delta t_{rec} \approx (1/f_{crit})$, with the flow exhibiting pseudo-recurrence behavior after every $\tau_{rec} \approx (1/f_{max})$. To construct the recurrence database, the full CFD-DEM simulation for a time period of 3 or 4 times τ_{rec} is utilized, ensuring that equilibrium is achieved.
The rCFD simulation of the fluidized bed was validated against the full CFD-DEM simulation and experimental data by comparing the mixing index for different fluidization velocities.
(image)
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Small-scale acoustic mixing of cohesive powders – challenges and solutions

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Abstract

Mixing of granular media is a fundamental unit operation in powder processing. The homogeneity of a powder blend is crucial for the product quality. One recent technology to achieve this homogeneity is acoustic mixing (AM), where the material which is to be mixed is subjected to vibration resulting in convective and diffusive flow within the powder bed breaking down segregations. AM gained a wider knowledge with the introduction of the resonant acoustic mixing (RAM) enabling various applications in pharmaceuticals, explosives and mechanochemistry. In addition to medium to large scale usage, AM is an effective option for mixing powder masses below 1 g in sealed containers. This small system size facilitates applications in material screening or, as in the present study, in personalized medicine. Tablets were produced in a single tablet scale direct compression process, where the ingredients for each tablet are dosed separately, mixed via AM and compacted to form a tablet. While the mixing process has proven highly efficient for free-flowing powder systems, challenges are observed for cohesive powder mixtures. This study presents the effects leading to these challenges and strategies to overcome them. High-speed imaging revealed the formation of a densely packed layer, for mixtures containing a relevant amount of cohesive powder. The stability of this layer depends on the frequency of the acoustic agitation. A systematic approach to identify suitable vibration parameters layer via standardized shear cell experiments preventing the formation of this stable was developed. This novel approach significantly improved the mixing quality for the cohesive excipients Sorbolac 400 and Vivapur 105 and opens opportunities to increase the processability of challenging powder systems.

Content

Atomic Force Microscopy Measurements for Guest-Induced Structural Transition Behavior of a Single MOF Particle

Prof. Satoshi Watanabe PhD; Homare Arima; Shotaro Hiraide
Kyoto University

Abstract

Flexible metal–organic frameworks (MOFs) exhibit S-shaped adsorption isotherms due to their structural transition. This guest-induced structural transition, so-called “gate adsorption”, shows a remarkable hysteresis loop between adsorption and desorption branches. Many studies have reported that the hysteresis behaviors changed depending on particle morphology of flexible MOFs, such as particle size and shape; however, the mechanism remains unclear. Revealing the dependence requires careful observation of the transition behaviors particle by particle. However, conventional adsorption measurements only address average behaviors over trillions of particles, and few techniques are available to investigate the transition behaviors at a single particle level. In this study, we propose a method for observing guest-induced structural transition of a single MOF particle using atomic force microscopy (AFM). In the AFM measurements, we gently pressed down on a single MOF particle to induce the structural transition and obtained corresponding force curves.
A thermodynamic analysis using measured force curves enabled capturing the transition behavior of a single particle.

Engineering supraparticles via spray-drying to achieve advanced functionalities

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Abstract

This talk will outline the possibilities and merits of designing advanced particles using the technique of spray-drying. Spray-drying can be used as tool to achieve forced assembly of nanoparticulate and / or molecular building blocks. Obtained are so-called supraparticles, i.e., micron-sized particles that possess a multimaterial composition and – along with a unique structure – yield new functional properties.
A brief idea of how structure control in such systems is possible will be given, followed by a demonstration of examples of highly functional systems that can be achieved, in particularly catalysts and “information-providing” particles.

Nanoparticles dispersion and aggregation behavior control for easily degradable adhesion under electrical pulse stimulation

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Abstract

Our research group has been investigating the relationship between the molecular structure of organic ligands and the dispersion behavior of nanoparticles in organic solvents and polymers using experimental and computer simulation approaches. In our previous studies, we experimentally determined the molecular structure of the ligands to disperse nanoparticles of various materials in various organic solvents and polymers. We also analyzed the relationship between the interface structure and dispersion behavior using molecular dynamics and discrete element method simulations. We apply our previous method of controlling the dispersion and aggregation behavior of nanoparticles to the separation process of composite bonding materials. By dispersing conductive nanoparticles such as silver and carbon black in strong adhesive raw material organic molecules for automobiles and other large equipment, we attempted to establish a method for easily decomposing adhesives by high-voltage electric pulse stimulation. The high-voltage separation process and mechanism by dispersing nanoparticles in adhesive bonding polymers were analyzed using high-speed video cameras and other methods.

Content

Analytical investigations related to agglomeration phenomena and its causes and mitigation

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Abstract

Optimizing Particle Size Distribution (PSD) of Active Pharmaceutical Ingredient (API) powders is essential for achieving a balance between bioavailability, stability, and processability in solid dosage forms. Storage and manufacturing processes of APIs typically do not occur under strictly controlled conditions, which can lead to agglomeration of cohesive micrometer-sized powders. These agglomerates can negatively impact processability, content uniformity, and bioavailability of the final drug product. A comprehensive approach to understanding the solid-state properties of APIs, with a focus on environmental conditions, helps identify their interdependencies and informs the execution of appropriate control strategies.

Utilizing time consolidation analytics with the ring shear cell (RST) and light microscopy (LM), combined with particle size distribution analysis by laser diffraction (PSD), allows for the detection and significance assessment of agglomeration. Additional techniques such as inverse gas chromatography (IGC) for surface structure and energy determinations, scanning electron microscopy (SEM) for particulate morphology, and X-ray powder diffraction (XRPD) for polymorphism state evaluation, provide deeper insights into the factors influencing agglomeration and guide preventive actions. This analytical approach is demonstrated through two case studies that evaluate strategies to ensure robust manufacturing processes and consistent pharmaceutical quality.

A Doe-Based Population Balance Model for Fluidized Bed Spray Agglomeration

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Abstract

Process modeling has undergone significant advancements over the past few decades. Despite the development in the field of particle technology, the design and analysis of particulate processes often remain rooted in empiricism. The need to represent these systems through a deeper understanding of the relevant phenomena and mechanisms has promoted a growing interest in model development. In this study, fluidized bed heteroagglomeration is described using an experimentally driven population balance model (PBM).

Fluidized bed agglomeration experiments with a top-spray configuration were carried out in a lab-scale plant (GF3, Glatt Ingenieurtechnik GmbH) using a pharmaceutical model material system: Avicel PH101 (microcrystalline cellulose) as excipient, sodium chloride as placebo substance (mimicking the API) and Pharmacoat 603 (hydroxyprpylmethylcellulose) as binder. The experimental design was structured using a Design of Experiments (DoE) approach. Key product parameters, including particle size, moisture content, API content, bulk density, and apparent density, were measured throughout the process. The collected experimental data facilitated the development of a population balance model capable of describing and predicting material transformations and process outcomes. The resulting model was implemented in the flowsheet simulation tool Dyssol [1], which allows the simulation of distributed properties of the produced agglomerates while considering the influence of the studied process parameters.

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Agglomeration in multistage spray dryer: A comparison of nozzle zone and fluid bed agglomeration

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Abstract

The standard set-up of spray dryer may be expanded to a multistage drying operation. In these the primary drying is completed in an integrated fluidized bed. This allows a more efficient drying especially in case where case hardening occurs. In these cases, particle size enhancement by agglomeration can be facilitated. Options include nozzle zone agglomeration, where the fines fraction is returned to the nozzle zone and agglomeration in the integrated fluid bed by its natural stickiness.

Fluid bed agglomeration can be subdivided into natural and spray on agglomeration. The spray on option is similar to the operation of standalone fluidized beds for agglomeration with the benefit of the integrated fluidized bed of being already in the fluidized state. The natural agglomeration by product sticky ness alone shall be discussed in detail. Especially the hypothesis that sticky point considera-tions related to the glass transition temperature can explain the agglomeration behavior will be sup-ported. This results in process control requirements like an on-line product moisture determination.

Both mentioned agglomeration techniques, Nozzle Zone Agglomeration and Integrated Fluid Bed Agglomeration, result in superior product properties as compared to standard drying. Particle size distributions and particle morphology are distinctly different as are powder flow.

Product characteristics are distinctly different to products ex nozzle zone agglomeration alone which has been studied intensively in the last years by several research groups.

Content

Short fibre reinforcement in Selective Cement Activation: Challenges of powder handling in the printing process

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Abstract

Selective Cement Activation (SCA) is a particle bed 3D printing process for concrete. The common raw material for printing is a dry aggregate/cement mixture, with aggregate sizes of below 1 mm. In the printing process, the parts are manufactured layer-wise, i.e. at first, a layer of the dry mixture is applied onto the build platform and compacted. Second, water is applied selectively onto the layer according to the construction of the part to be build to activate the cement in the specific regions . These steps are repeated, until the part is finished. Afterwards, the part needs to be excavated from the particle bed. A challenge of the process is the low strength of the printed parts. In conventional concrete technology short fibres are added, to enhance the tensile strength. There are already studies showing a beneficial effect of short fibres in SCA. However, the material was applied manual to the build platform.
In order to investigate the suitability of fibre containing mixtures for automated SCA material application, we tested the influence of the fibres on the bulk solids properties. Therefore, aggregate/cement mixtures containing short fibres were produced and the volumetric fibre content, fibre material, fibre thickness and fibre length was systematically varied. The mixtures were tested for bulk density, tap density, flowability, dynamic angle of repose as well as their elastic spring-back after normal load. It was found, that especially the fibre material influences the bulk solids properties of the mixture: Less ductile fibres lead to a smaller impact on the bulk solids properties. Finally, specimens were printed with the most promising mixtures, to investigate the influence on the printing process and the mechanical properties of printed specimens.

Content

Under(-)pressure – Analyzing the driving forces behind the die filling under suction on rotary tablet presses

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Abstract

Tablets are the most commonly used dosage form. The filling process is considered to be a critical step in tablet production as it influences quality attributes and is not yet fully understood. The commonly used filling mechanism for single-layer tablets is suction filling, for which the lower punch is pulled down below the powder bed inside the feed frame. This pull-down movement creates a differential pressure leading to an assisted powder flow into the die. In the past, it has been investigated how the die filling efficiency is related to the pull-down velocity of the lower punch, as well as how the powder flow through an orifice is related to the applied differential pressure. As a novel approach, the goal was to analyze the pressure gradients as a result of the pull-down movement. Therefore, a 3D-printed component was designed for airtight sealing of the feed frame filling area of a rotary press. Openings were installed at defined points determined by the design of the fill cam, to which a differential pressure sensor could be connected. The developing negative pressure could be measured in a setup using a feed frame without powder along the pull-down and dosing angles at various turret speeds. These results can be used for direct correlations between the forces acting as an effect of the differential pressure and the filling performance for materials with different permeabilities. Based on the findings regarding how the pressure conditions in the die behave along the fill cam and the dosing unit, it is possible to derive more precise statements about the forces and accelerations acting on the powder during die filling. Additionally, this will allow the optimization of the design of fill cams and dosing units and their positioning in relation to the opening angle.

Content

CFD-Simulation of CO2 adsorption for carbon capture in a packed bed based on geometry creation by the Discrete Element Method

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Abstract

Carbon Capture is an important technology for the reduction of CO2 emissions in industrial processes. An example is cement production, which produces CO2 as a reaction product. A common separation process is amine absorption, which has a high energy demand during regeneration. The usage of solid adsorbent promises the reduce of energy demand.
The formulation of the carrier was tested by tableting and granulation leading to longish cylinders, flat cylinders or spheres. The inner surface was determined by N2 adsorption in BET. The formulation step strongly influences the porosity, stability and surface.
Too high compacting pressure showed to result in capping of the tablets, resulting in the best results at a medium compacting pressure. The surface showed to be highest at lower compression, which was to be expected. The surface is highly dependent on the agglomeration mechanism, which is visible in SEM. Different approaches are under investigation to determine diffusion coefficients from BET raw data of CO2 adsorption tests.
To simulate the adsorption process, the packed bed was created using the discrete element method, exported, smoothed and meshed. The process leads to a smoother geometry than the real packed bed, but creates a realistic settled packed bed. The adsorbent zone is represented as a porous zone. The adsorption process is modeled in CFD by the convective and diffusive transport of CO2 between adsorbers, and also diffusion to the inside reaching a capacity limit. This approach reproduces the capture of CO2 first to the surface and by diffusion through pores to the inner region, reproducing breakthrough curves. The size is limited to several gram of adsorbent due to the small local mesh size.

Content

Spreadability vs. Flowability: Measurements of metal powders for additive manufacturing

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Abstract

Additive manufacturing (AM) of metals is primarily used in lightweight construction and prototyping in various industries (automotive, aerospace and medical industries). A frequently used process in the additive manufacturing of metals is the powder bed fusion with a laser beam (PBF-LB/M). In PBF-LB/M, a powder bed is applied layer by layer to a build platform and selectively melted with a laser, building up the final part. The spreading of powder is conducted with blades and/or rollers. To produce dense components, it is essential to apply a homogeneous and dense powder bed. Due to the low acting normal stresses and the dynamic nature of the spreading process, conventional flowability measurements can only be taken into consideration with limitations. It has not yet been possible to transfer conventional flowability to spreadability. In this article, the flowability is therefore measured at very low stresses conventionally and is compared with measurements using a spreadability test rig.
To gain a more elaborated understanding of flowability and spreadability (and their differences) the model material Stainless steel powder (1.4404) is also coated with nanoparticles (i.e. silicon carbide) to alter the surface roughness. Conventional bulk solid properties are characterised using a ring shear tester (at very low normal stresses) and dynamic measurement methods (e.g. dynamic angle of repose). The spreadability of metal powders is investigated using a spreadability test rig. Here, the powder is spread into a sample volume, which is then analysed using X-ray microtomography (µCT). In this way, influences of powder properties, blade geometries, blade speed and spreading distances on the powder bed structure are investigated and discussed in relation to the flowability.

Content

Modification of Powder Properties by Liquid Additives

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Ohm Technische Hochschule Nürnberg

Abstract

Additives are applied to dry, fine powders to achieve a wide range of objectives, including the reduction of energy consumption, refinement of particle size distributions, enhancement of product throughput or dosing behavior. These modifications often result in improvements in flowability, a reduction in agglomeration tendency, decreased frictional resistance, and the development of anti-stick properties. While numerous individual studies have investigated various additives across diverse processes, many results remain fragmented, and a comprehensive understanding of the mechanisms underlying the actions of different additives is still lacking. Consequently, the selection of suitable additives continues to rely predominantly on empirical knowledge.
In this work, the addition of liquid additives from various substance classes to powders was investigated in greater detail. In particular, the effects of additive incorporation on the behavior of powders during interactions with other materials were examined—whether during the admixing of additives in different mixing systems or upon contact with various surfaces in the fluidized state, using a specially developed experimental setup. While simple geometries and varying process parameters can be tested in the experimental rig, practical testing of the additive products was also conducted in more complex systems. Simultaneously, other properties—e.g., changes in surface energy, dusting propensity, or agglomerate strength—were also studied. The results obtained aim to broaden the understanding of the effects of specific additive classes on products from different perspectives, thereby increasing the transparency and soundness of the additive selection process for diverse future applications.

Content

Powder rheological characterization of dry battery electrode materials

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Abstract

The novel dry battery electrode (DBE) process has advantages compared to established solvent-based coating procedures including environmental benefits, reduced costs and enhanced battery performance. In the DBE process, the active material, conductive additive, and polytetrafluoroethylene (PTFE) binder transition from fine powder to the free-standing electrode under high shear and elevated temperature, enabled by PTFE fibrillation. This is critical for processability and the final battery performance, requiring a homogeneous mixture with the appropriate fibrillation before calendering. The goal of the study was to apply conventional powder rheology for characterizing the powder behaviour during this innovative battery production route as well as to develop a new method to characterize the fibrillation process of the PTFE binder.

During the first part of the study, deaeration time, segregation, shear and compressibility testing were applied. For this purpose, mixed anode materials (graphite, carbon black, PTFE) were characterized both before fibrillation (as premixes) and afterwards. The binder content played a significant role on the segregation where a higher binder proved to prevent it. Interestingly, the powder's flowability was increased for the premix with the highest binder content with increased applied normal stress.

The second part of the study focused on developing a method for characterizing PTFE fibrillation. Wall friction measurements with an underlying temperature ramp revealed a strong increase in shear stress only once PTFE's crystalline transition temperature below 20 °C was transgressed (which is the prerequisite for fibrillation). This was correlated with the PTFE's fibrillation by SEM pictures taken at the different stages of the wall friction test.

Content

Radiation-based techniques for detailed investigation of cohesive particle flows

Prof. Dr. J. Ruud van Ommen PhD; Kaiqiao Wu; Rens Kamphorst; Christian van der Sande; Evert Wagner; Dr. Gabrie M.H. Meesters
TU Delft

Abstract

There is an increasing interest in ultrafine particles. Such particles are often difficult to process because of their cohesive nature. Similar problems are encountered for wetted particles. This means, for example, that a regular fluidized bed does not provide sufficient mixing, but that additional methods such as stirring or vibration are needed. In recent years, we have developed X-ray imaging and single-photon emission radioactive particle tracking to obtain detailed information about gas-particle flows. In this work, we will discuss some examples of obtained results.

The X-ray setup consists of a triplet of cone-beam sources (X-ray tubes) and three detector panels. For imaging, we often use only a single source and detector panel. The X-ray imaging reveals that for a fluidized bed of micro-silica, vibration reduces channeling, but does not significantly change the agglomerate size. When using a stirrer to enhance the powder mixing, the design of the stirrer is of large influence: it should maximize the sweeping coverage and avoid the creating of gas pathways. When required, the three sources and panels can be used to make a full 3D tomographic reconstruction of a system under study.

While the X-ray imaging gives precise information about the density distribution, single-photon emission radioactive particle tracking can be used to obtain the trajectory of a representative particle. We use a polystyrene bead with an activated gold core – emitting gamma-rays – as the tracer particle. Three identical γ-radiation slit collimator detectors are placed equidistantly around the system of interest. We demonstrate for a horizontal stirred bed that increasing the agitator rotation speed and the fill level of the bed both lead to an increase in the solids circulation.

Content

Powder Mixing and Segregation in a Cylindrical Bladed Mixer

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Abstract

Fine-grained particulate matter, which is an inevitable output of many industrial processes, causes a variety of challenges. These different residues can be processed in an optimized manner to generate value and to minimize hazards. This involves many opportunities for the application of bulk powder technologies for sustainable products, new analytical methods as well as modelling and simulation.
One approach in the Helmholtz project FINEST is targeted merging of material streams that are extremely different in terms of chemical and phase composition. This involves a mixing and agglomeration step, followed by pyrometallurgical treatment.

In powder mixing, differences in particle properties cause segregation. We focus on different densities, representing a range of residual fine particles from plastics over minerals to metals. We choose a non-invasive technique to investigate the process, namely Microfocus X-Ray Computed Tomography (µCT). Combinations of particles from 150 – 250 µm with densities between 0.5 – 5 g/cm³ are mixed in a cylindrical bladed mixer. The µCT scans of powder mixtures are acquired with voxel size of 100 µm. They provide insight into the composition of the powder mixture at any point in space and allow to analyse the powder flow. Using a 3D analysis method, the quality of mixture is described by a variance measure. The method features a direct analysis of intensity/grey scale values from the µCT data. It needs no segmentation or binarization step. Instead, a normalization is applied based on histogram analysis. The known principle of sampling bulk powders and sample statistics are adapted. In analogy to physical samples, grid cells are constructed over the image data. Within those, a mean intensity value is determined and variance of those mean values

Content

calculated. The evolution of variance describes the evolution of mixing quality over time. A mixing index, following classical variance based mixing index approaches, can be formulated on this basis.
The method is applied to a variety of mixtures. We provide a full image of parameters, such as density ratio, mixing speed and mixing duration that lead to segregation and the dependency of the variance measure on those.

Complementary, we perform simulations. To this end, continuum modelling is chosen to simulate the mixing. We are currently implementing μ(I)-rheology and a density segregation term and validating it against the experimental results.
It combines previous continuum models for bladed mixer considering only size-segregation, other geometries being investigated for density segregation and implementations of the rheology in free surface flows.

Acknowledgement: The project FINEST is funded by the Investment and Networking Fund of the Helmholtz Association under grant agreement no. KA2-HSC-10.

Modelling of nitrogen transport and loss during the additive manufacturing of martensitic stainless steels

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Abstract

Martensitic stainless steels (MSS) based on the FeCrC alloy system are known for their high strength and excellent corrosion resistance, which are primarily attributed to a chromium (Cr) content greater than 13 wt.%, with at least 10.5 wt.% dissolved in the steel matrix. Strength is predominantly determined by the carbon (C) content and the martensitic microstructure in the quenched and low-tempered state. To simultaneously enhance both strength and corrosion resistance, high nitrogen (N) contents above the maximum solubility are added. Moreover, the interaction between C and N further strengthens their individual effects, making N a key alloying element for MSS, with additional sustainability advantages as it is an abundantly available resource. Despite these benefits, introducing nitrogen into MSS presents challenges. Nitrogen solubility in molten steel is significantly lower than in the solid state, and exceeding the nitrogen solubility limit during the melt stage of conventional metallurgical processes can lead to nitrogen outgassing and the formation of gas porosities. Powder bed-based additive manufacturing techniques such as Laser Powder Bed Fusion (PBF-LB/M) are of increasing interest for producing high-performance materials. In this process, fine metal powders (typically 20-60 µm) are selectively melted using a focused laser, layer by layer, enabling the creation of complex geometries without the need for specific tools or subtractive manufacturing steps. The high solidification and cooling rates prevent the nitrogen to reach its equilibrium state resulting in nitrogen contents above the maximum solubility. During PBF-LB/M it remains challenging to observe the nitrogen outgassing behaviour and the transport inside the metal phase. In the past numerical approaches have proven to be successful in revealing the multiphysical transport phenomena inside the metal phase. Thereby, this work couples a DEM-FVM method to investigate the impact of energy density during PBF-LB/M onto the nitrogen content inside the X30Cr15N (0.7 wt.% N) alloy. The DEM is applied to solve the spreading behaviour, afterwards the powder bed can be exported into the FVM to model the PBF-LB/M process of single tracks. The FVM solves transport equations for mass, momentum, heat including a laser ray tracing algorithm, and the nitrogen mass fraction. The numerical results for the nitrogen content and the melt pool size are validated based on experimental measurements. The final results indicate that the nitrogen degassing increases for higher energy densities.

Content

Better structural color with the help of a computer

Prof. Nicolas Vogel
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Abstract

The beautiful coloration observed in the natural world is often based on the interaction of light with defined nanostructures within butterfly wings, bird feather, insect exosceletons, etc. Such colors can be recreated by mankind using self-assembly. In this respect, colloidal particles are interesting tools, as they can be synthesized with high control of sizes and be self-assembled into desired nanostructures. However, self-assembly inherently produces defects, which can compromise the observable color as random scattering is superimposed on the photonic properties of the material. Broadband absorbers are often added to increase the color saturation. In the natural world, this absorber addition is realized by incorporation of melanin, which can be synthetically mimicked by polydopamine. While this strategy has been applied to create more vivid structurally colored materials, little is known about the optimal amount of absorber, nor about the best possible position to add this absorber within the colloidal photonic crystals.
Here, we use a predictive design approach to optimize structural coloration. We use mathematical structure optimization to let the computer produce blueprints with ideal structural arrangements that produce structural color with the highest possible chroma. We experimentally control the self-assembly process to realize these structural predictions and investigate the color properties as a function of absorber content and position.
The potential of predictive design reaches beyond the scope of mere chroma optimization and may provide new strategies to optimize the properties of particulate systems more generally, provided that the physics underpinning the size- or shape-dependent properties are established.

Content

Continuous flow synthesis of Au nanoparticles directly combined with chromatographic separation

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Abstract

Small gold nanoparticles (Au NPs) with diameters < 5 nm are interesting in catalysis due to their large surface-to-volume-ratio. Their production requires high nucleation rate and fast reduction of the Au precursor and is therefore a mixing controlled process. Traditional batch processes suffer from limited reproducibility and scalability as mixing is hard to control. In contrast, continuous flow processes provide well defined mixing and proper adjustment of the flow and local energy dissipation by varying the Reynolds number (Re). We studied the influence of Re in a T-mixer on the particle size distribution (PSD) of Au NPs and found that the mean particle size and the width of the PSD decrease with increasing Re, whereas above a critical value of Re the PSD becomes independent on Re as the mixing control is overcome. Additionally, the Au NP formation process was studied by quenching the Au NP growth at certain time steps to identify emerging intermediate species. We developed a Finke-Watzky model based on population balance equations. The model includes the reduction of the Au salt and the growth of the Au NPs by agglomeration and provides nearly perfect agreement with the experimentally determined time dependent PSDs.
In spite of optimized Au NP syntheses, often a subsequent classification step is required to fine tune the particle properties for certain applications. We demonstrate how the continuous flow synthesis of Au NPs of different size and PSD width can be directly combined with chromatography. We discuss the potential of a direct coupling of the continuous Au NP synthesis with size exclusion chromatography for purification of the Au NPs from side products and for narrowing down the PSD of the Au NPs by classification in different size fractions.

Content

Silver-lined gold patches: Continuous flow synthesis of particles with long-term stable visible and infrared resonances

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Abstract

Patchy nanoparticles combine a dielectric core with a partial noble metal coating, enabling localized surface plasmon resonances (LSPR) that can be tuned more broadly than in simpler isotropic systems. However, these anisotropic structures often suffer from morphological instability, which limits their application. In this study, we demonstrate a continuous triple T-mixer flow process that produces robust silver–gold patchy nanoparticles on cationic polystyrene nanospheres in three distinct steps: (1) small gold seeds attach via heterocoagulation, (2) dense gold patches grow via electroless plating, and (3) silver ions are introduced under mild reducing conditions to overcoat the patches. Scanning electron microscopy, high-angle annular dark-field scanning transmission electron microscopy, and energy-dispersive X-ray spectroscopy reveal that silver preferentially deposits on the gold regions, resulting in bimetallic patches with independently tunable coverage (governed by gold precursor amounts) and thickness (determined by gold and silver precursor amounts). A simple geometric model links the evolving patch aspect ratio to LSPR shifts, clarifying how the amount of each metal precursor influences the resonance shift from visible to near-infrared wavelengths. Notably, a silver content of ~15% preserves patch morphology for at least four months by effectively reducing the surface energy, while lower silver amounts prove inadequate, and higher loadings lead to partial re-dissolution. These findings elucidate how balancing bimetallic composition and surface coverage can maintain anisotropy and stabilise optical properties. The resulting process effectively bridges the gap between specialised nanostructures and large-scale, application-tailored production.

Content

Kinetic Monte Carlo Simulations for Nanocrystal Shape Control

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Abstract

The precision of nanocrystal shapes is crucial to tailor the functionalities of nanomaterials. Traditional molecular dynamic simulations are computationally too expensive to unveil the multiscale nature of nanocrystal synthesis from the potential energy of an atom to the mesoscales of a nanocrystal composed of tens of millions of atoms. To overcome this issue, we implement rejection-free kinetic Monte Carlo simulations using the semi-Gibbs ensemble sampling solid-to-liquid energy variations to grow and dissolve atoms at the nanocrystal surface. This allows the simulation of realistically sized nanocrystals coupled with the energetics of atoms. We discuss the growth of symmetry-preserving shapes, such as cubes, octahedra, rhombic dodecahedra, and their truncations. We show the importance of surface site kinetics associated with adatom nucleation on facets of different crystallographic directions, leading to the entrapment of nanocrystal shapes in metastable equilibrium. We then discuss the spontaneous symmetry breaking of shapes due to the dynamics of surface defects. The multiscale simulations reproduce the emergence of nanocrystal shapes inaccessible to other computational tools.

Content

Gentle, sustainable and innovative encapsulation and drying for new powder

Jerome Vallejo
Lis By Lesaffre

Abstract

Drying is one of the processes allowing a better conservation of food or organic product in general. It is acting directly on the availability of water in the product, which is a good way to limit or to block the development of contaminant such as bacteria and mold.
The key factors impacting the quality of the product obtain during the step of drying are: the temperature of the product during the drying, the time during while the product will remain at this temperature, the contact with the drying air for oxidation phenomena for instance. Will see how spray drying allows a very fast drying of a liquid product and the production of a dry powder in one sustainable step. Vacuum belt drier is another type of drying performed under vacuum; the temperature of the product can be very low. This type of drying is a conducting drying and thus airless. Extrusion Porosification Technology and POLARDRY are new alternatives to drying technology. EPT allows to work in a more optimized way from regular spray drying by combining extrusion and spray drying. POLARDRY uses a polarization of the spray in a neutral atmosphere (N2). New development such as FlowFocusing gives a new way to encapsulate sensitive component: this disruptive technology allows to produce in one step core-shell dried capsules.
LIS is a partner with the know-how on technologies such as Spray Drying, Vacuum Belt Drying, EPT, POLARDRY and FlowFocusing to help you in creating and improving your products. We also have a laboratory in Rennes (Powder Studio) to characterize the powder obtained in order to evaluate, compare and improve different type of powder (shape and/or formulation).

Content

Reconditioning of Spent Graphite from End-of-Life Lithium-Ion Batteries through High-Temperature Treatment

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Abstract

Recycling spent batteries represents a sustainable approach to prevent material shortages and the waste of valuable resources in the expanding lithium-ion battery (LIB) market. Previous research has focused on cathode materials, while graphite, the most commonly used battery anode material, is often discarded. However, to reach EU regulation targets graphite must also be recycled. This study proposes a novel thermal reconditioning process for graphite.

The spent graphite was obtained from aged battery cells through a series of processing steps, including comminution, classification, pyrolysis and leaching. Nevertheless, the graphite still contains impurities (up to 4 % by weight) and structural or morphological defects, e.g., an increased specific surface area. Some of the impurities adhere to the surfaces of the graphite particles reducing their specific discharge capacity. To reverse the aforementioned changes, the spent graphite is subjected to a high-temperature treatment at various temperatures (2000 °C to 3000 °C). With increasing temperature the amount of residual amorphous carbon decreases. Moreover, any residual metallic impurities are effectively removed. Both effects contribute to a reduction in the specific surface area.

The reconditioned graphite exhibits good electrochemical performance at low charge rates, highlighting the importance of high graphite purity. An increase in charge rates, however, results in a notable reduction in the specific capacity due to the removal or alteration of the previous surface modifications of the graphite particles. Consequently, a pitch-based coating is applied to enhance the fast charging capability. This study enables the reuse of graphite in LIBs, paving the way for the industrial implementation of graphite recycling.

Content

Influence of defects and mechanical damage on the antifouling behaviour of Slippery Liquid-Infused Porous Surfaces (SLIPS)

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Abstract

The liquid repellent properties of superhydrophobic surfaces and SLIPS coatings have been promising for applications in antifouling and anti-icing coatings as well as self-cleaning materials . We have recently explored sustainable coatings with efficient repellency properties . They rely on renewable materials, fluorine-free components and water-based fabrication processes . In particular, we used an aqueous dispersion of binder polymer and hydrophobic fumed silica particles. The dispersion was applied on various substrates by spray coating. Upon drying, the particles self-organize into a micro- and nanostructured thin film with superhydrophobic properties. This porous film is then infiltrated with silicone oil to achieve liquid-infused characteristics with efficient repellency. This can protect the surface from multiple forms of fouling, including corrosion and microbiological fouling. But as in every surface protective coating, scratches remain as a threat for the systems repellency properties, which may compromise long-term stability in real world applications.
We hypothesize that liquid-infused coatings may be advantageous in this regard, as the self-healing properties due to the presence of fluid lubricant within the surface, which remains mobile and can re-cover defects. We explore this effect of mechanical damage in a set of self-made and industrial SLIPS-based coatings in their performance against biofouling in freshwater systems. Mechanical damage was imposed by scratching with a tungsten carbide needle and the effect on the corrosion prevention explored by a set of different techniques, including electrochemical testing and microbiological fouling.
The application was also tested in field studies under real-life conditions.

Content

DEM-CFD Study on the Impact of Cross-Shaped Internals on Biomass Drying in Rotary Drums

Alina Clara Lange; Prof. Dr.-Ing. Harald Kruggel-Emden
Technische Universität Berlin

Abstract

1 Introduction

Thermal drying is often the most time- and energy-intensive step in the material and thermal utilization of wet biomass [1]. Due to the heterogeneity of wet biomass, convective dryers have become the most commonly used drying method, with the drying medium playing a major role in heat transfer [2]. Lifters are often installed inside the drum to facilitate the cascading and distribution of the material, increasing particle contact with the main gas stream and thus improving heat and mass transfer [3]. This interaction is critical to the drying progress and the overall performance of the dryer. This study examines the impact of cross-shaped internals additionally installed to the lifters on the transverse movement of wooden biomass within rotary drums. The goal is to extend particle residence time in the central drum region, enhancing contact with the primary air stream and improving drying efficiency.

2 Numerical methods

A coupled DEM/CFD modeling framework was developed to simulate the particle drying process. The discrete element method (DEM) is used to model particle dynamics, while computational fluid dynamics (CFD) captures the fluid phase and particle-fluid interactions. This integration enables a detailed investigation of heat and mass transfer mechanisms governing drying behavior at the particle level.

To accurately describe the drying process, both the heat balance of individual particles and the variation in moisture content—governed by mass conservation of the evaporating component—must be considered. Given that the operating temperature remains below 700 K, radiative heat transfer is neglected [4]. Thus, the model considers only the latent heat associated with water evaporation, as well as convective and conductive heat transfer between particles and between particles and walls.

Content

The convective heat transfer rate between the gas and particle phases is modeled using Newton's law of cooling, expressed as the product of the surface area of the interface between solid and drying medium, temperature difference, and the convective heat transfer coefficient. This coefficient is estimated via the Nusselt number, which is computed as a function of the particle Reynolds number and Prandtl number using the Gnielinski correlation [5] realizing for varying void fractions.
The latent heat of evaporation is determined as the product of the enthalpy required to remove moisture from the solid and the rate of moisture evaporation. The latter is modeled using the reaction engineering approach proposed by Chen et al. [6].

3 Simulation setup

Simulations were carried out for a drum rotation speed of 15 rpm and a filling level of 15%. The particles are spheres of beech wood, with a diameter of 10 mm and an initial moisture of 0.76 (kg water/kg dry wood). The inlet air has a velocity of 2 m/s and a temperature of 130 °C.
The geometry selected for this study is a rotary drum with a diameter of 500 mm and a length of 150 mm, in which different internal configurations were installed along the entire length of the drum. The first configuration (0 L + 0 I) corresponds to an empty drum, which was used as a reference to evaluate the effect of internal components on particle drying. In the second configuration (10 L + 0 I), ten lifters with an axial and radial length of 50 mm were uniformly distributed along the drum. The third configuration (10 L + 1 I) includes ten lifters and a single internal cross. In this setup, the center of the cross is aligned with the center of the drum, and each of the four arms has a length of 150 mm. The final configuration (10 L + 4 I) includes ten lifters and four internal crosses, uniformly distributed in a circular arrangement with a radius of 120 mm, and each cross has a length of the four individual arms of 70 mm.

4 Results

One of the key parameters analyzed to evaluate drying performance across the different geometries was the distribution of moisture content (X) among individual particles. This parameter is crucial, as a more homogeneous moisture distribution indicates more uniform drying, which helps prevent issues such as over-drying or under-drying that can affect product quality and energy efficiency.
To assess particle moisture content in the simulations, the distribution of X for each particle was obtained after one complete rotation of the drum for all four geometries.

The results show that in configuration 0 L + 0 I, the X distribution is more dispersed, with most particles retaining higher moisture levels compared to the other configurations. Configurations 10 L + 0 I and 10 L + 1 I exhibit similar trends, although configuration 10 L + 1 I features a greater proportion of particles with lower X values. Configuration 10 L + 4 I shows the narrowest distribution, with a peak at the lowest moisture levels among all cases, highlighting the effectiveness of multiple cross-shaped internals in enhancing drying and promoting a more homogeneous moisture distribution.

5 Conclusions

Using the coupled DEM/CFD framework, the performance of different internal structures is evaluated by analyzing particle-scale moisture and temperature distributions, as well as comparing drying rates. The results highlight the strong influence of internal geometry, operating conditions, and parameter variations on drying efficiency. These insights provide valuable guidance for optimizing rotary drum design, supporting improvements in energy efficiency and drying performance.

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Chromatographic fractionation and characterization of functional nanoparticles

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Abstract

Scalable techniques to produce nanoparticles (NPs) typically lead to property distributions in terms of size, shape or composition. Therefore, a classification step is mandatory to tailor the synthesized particle ensemble according to the requirements of the product. We demonstrate that NP chromatography is highly promising and scalable or fractionation and characterization. Specific targets for classification are: Separation of a broader feed into two or more fractions, the removal of fine or coarse tail fractions, shape-selective separation, or the separation of by-products. We study the classification of different NPs in the size range from 1 nm up to 100 nm. Examples are the separation of C60 from C70 fullerenes, carbon nanodots, high precision classification of semiconducting quantum dots with respect to their bandgap, semiconducting gold clusters and plasmonic gold and silver particles and their alloys.

Size exclusion chromatography relies on repulsive interactions between the stationary phase material and the NPs, which can be tailored by applying concepts from colloid science via DLVO theory. Remarkably, the fractionation is independent of the type of material as shown for Au, Ag, alloys, SiO2 and polymer NPs. This is the basis to measure accurately even multi-modal size distributions and supports two-dimensional shape characterization in a single separation step.

Via interaction-based chromatography, we show how to fractionate gold nanoclusters. In particular, hydrophilic interaction chromatography, which uses organic solvents, is replaced by purely water-based anion exchange chromatography as a sustainable and scalable alternative. Options for scale-up are discussed. Finally, preparative chromatography is coupled with continuous nanoparticle synthesis.

Content

Production of supraparticles using anisotropic nanoparticles via single droplet drying

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Abstract

In this work, the morphology of supraparticles using nanosuspensions of anisotropic particles is investigated. A detailed analysis of the drying kinetics for the particle formation process, especially the locking point and size, is conducted via single droplet drying experiments. The influence of material conditions and process parameters, namely solid content in the droplets and temperature, on the supraparticle structure formation is experimentally investigated and discussed. Furthermore, quantitative characterization of the dried supraparticles (size, crust thickness, and packing structure) is done via Electron Microscopy.
We gratefully acknowledge the financial support of the Deutsche Forschungsgemeinschaft (DFG – German Research Foundation) within the collaborative research center 1411 - Design of Particulate Products

Synthesis of ultrafine metal oxide nanoparticles using continuous flow hydrothermal reactions

Dr. Akira Yoko PhD
Akira Yoko

Abstract

Supercritical hydrothermal synthesis is the methodology of synthesizing metal oxide nanoparticles through hydrothermal reactions using a continuous flow reactor. Moreover, organic compounds and water can form a homogeneous phase at high temperature compressed conditions, which enables the in situ organic modification at the same time as the particle formation. Continuous flow synthesis is advantageous for scaling up as well as the precise control of the products. In this study, fine control of ultrasmall particles (e.g., < 5 nm) was achieved for metal oxides, which was difficult to synthesize previously.

Cerium octanoate complex precursor was used as the precursor of supercritical hydrothermal synthesis. Synthesis of octanoic acid modified CeO2 nanoparticles was conducted using a continuous flow reactor. The reaction temperature was set to 300 or 340 °C. The reaction time was set to 0.04 to 380 s by changing reactor volume.

The distribution of particles was narrow as < C.V. 0.25. The ultrasmall particles including < 2 nm were obtained. Significantly, the conversion was more than 99% at the shortest reaction time of 0.04 s, and almost all precursors were converted to particles. Moreover, the crystallinity was surprisingly high even with small domain size. By increasing reaction time, particles grew up to 3〜5 nm rapidly, and after that, slow growth as usual ripening was observed. The initial rapid growth was assigned as fusion growth with different growth rate compared to the ripening at the later stage. The fusion growth, which can be regarded as nonclassical particle formation, was verified with detailed analysis of TEM and synchrotron XRD. Furthermore, the behavior of the obtained nanoparticles as nanofluids and as nanocatalysts were studied.

Japanese Process Science Project Toward Design and Control of Nanomaterials

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Tohoku University

Abstract

In order to realize fine control and design of nanomaterials and promote thier social implementation, “Process Science” project to build a platform for nanomaterials engineering had been conducted from 2019 in Japan. In this project, based on thermodynamics and chemical engineering, phase equilibrium behavior (dispersibility and aggregation) prediction based on nanoparticles as pseudo-molecules, construction of prediction equations for nanofluids’ transport properties (diffusivity, viscosity, thermal conductivity), and design of unit operations (classification, separation, mixing) for nanomaterials have been realizing. Furthermore, knowledge has been accumulated on the prediction and design of structure formation of nanomaterials, which is outside the scope of conventional chemical engineering.

Decoding breakage mechanism under impact and compression in slags from pyrometallurgical recycling of Lithium-ion batteries via mineral liberation analysis

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Abstract

The increasing demand for lithium-ion batteries underscores the need to develop efficient recycling processes that can recover valuable metals, particularly lithium, while addressing environmental concerns. This study explores the breakage mechanisms of slags derived from the pyrometallurgical recycling of end-of-life batteries, with a particular focus on lithium-containing phases. To this end, thermodynamically designed synthetic slags and a battery slag were used to investigate the influence of stress types (impact and compression) applied by various milling and breakage investigation methods. The primary objective was to determine whether the breakage of these complex materials is a random event or could be strategically influenced by the slag's inherent properties, such as mineralogical composition, crystal size, particle size, and the energy applied during milling processes. The mineral liberation analysis, facilitated by scanning electron microscopy, served as the primary technique for identifying and quantifying the microstructure and thus different phases present in the slag and their breakage. Furthermore, a tool has been developed to analyze images obtained from mineral liberation analysis. This tool utilizes machine learning methods available in Streamlit's open-source app framework. It facilitates a more dynamic and interactive approach to data interpretation and phase categorization. Based on both analyses, the impact stressed particles showed a better enrichment of eucroptite (x50 < 100 µm).
By understanding of the relationship between the applied stress and the resultant particle size and phase distribution, it is possible to improve the liberation and recovery of lithium from recycled battery slag.

Content

Influence of compaction kinetics on dynamics-dependent material deformation and machine artefacts

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Abstract

In the production of pharmaceutical tablets, it is essential to accurately meet critical quality attributes, including strength, disintegration time and release kinetics, which are influenced not only by compression stress but also by compression dynamics. Based on material's physicochemical and mechanical properties, its deformation behaviour may depend on speed of loading and unloading as well as the time of maximum loading. This introduces a potential conflict of objectives with economic production on rotary presses, especially when the formulation relies on kinetic-sensitive materials, such as active pharmaceutical ingredients (API). In consequence, high compression speeds may result in impaired product quality or destructive defects like capping or delamination.
In this study, dynamics-dependent material deformation and dynamics-dependent machine response are intensively studied, stressing the need for a differentiation of kinetics-dependent signal artefacts. To investigate this topic, experiments using a compaction simulator (CS) are presented. Increasing compression dynamics can result in artefacts due to the construction of the CS and can disturb and distort the measurement signals. It is therefore necessary to identify the extent and origin of the signals and to develop a method to deconvolute the dynamics-dependent material deformation signals from such artefacts resulting from dynamics-dependent machine response. Based on this, descriptors for the compression speed dependence of materials are deduced from corrected data.

Content

Improving Particle Morphology in Fluidized Bed Spray Granulation

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Evonik Operations GmbH

Abstract

Fluidized bed granulation is a well-established unit operation extensively employed for the production of granular particles from solutions or suspensions, leveraging the efficient heat and mass transfer characteristics inherent to fluidized beds. The physical and mechanical properties of the resultant particles directly influence the quality and specifications of the final product, as this process typically represents the last production step that determines these attributes. Key particle characteristics, including shape, surface morphology, internal structure, as well as bulk and particle density, are established during this phase and are closely associated with functional properties such as moisture content, caking behavior, and particle size distribution.

This overview examines an existing high-throughput fluidized bed spray granulation system, providing insights into the mechanisms governing the development of the internal structure of granules produced within a fluidized bed. The discussion focuses on the strategic design of compact particles through the targeted manipulation of the sprayed liquid and associated spraying parameters. Furthermore, modifications to these parameters have been shown to significantly improve overall equipment availability. Improved coating and drying efficiency of the particles resulted in reduced caking, thereby increasing operational uptime between cleaning cycles.

Content

DEM Simulation of Roller Mill Feeding and Performance

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Abstract

The roller mill as a comminution machine has garnered increased interest due to its ability to achieve a controlled product size distribution through single-particle fracture with precise compression ratio control. Research by Schönert and others has shown that slow compression of single particles is the most energy-efficient breakage mode, outperforming impact, attrition, abrasion, and confined interparticle breakage. In a fixed-gap roller mill, a feed particle fractures if its size is smaller than the roller gap. Particle shape also plays a role, as the dimension determining passage through a slit differs from that for classification through a square hole. Thus, particle size distribution in roller mills must account for this deviation, which also impacts particle shape modeling discussed later in the paper.
If the feed is controlled to ensure limited particle interaction, a single-particle breakage mode can be maintained. The term “gap occupancy” refers to the volumetric ratio of particles in the gap relative to capacity. Analytical models provide qualitative insights into mill performance but lack detailed understanding of the milling process. While DEM simulations with particle fragmentation are still evolving, they reveal specific limitations of mill behavior.
This paper presents DEM modeling and simulation techniques, incorporating a GPU-based dilated polyhedral discrete element method with a cohesive zone fracture model. Particle interactions with rollers generate global reaction forces and torque. Repeated contact forces lead to local surface fatigue and roller wear.

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Screw design in decanter centrifuges by experiment, Discrete Element Method and Computational Fluid Dynamics

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Abstract

The olive oil extraction process typically involves milling olives, malaxation and subsequent separation within a decanter centrifuge. Approximately 5 to 15% of the oil is lost in the separation step. Presented is the redesign of the decanter to break the pomace cake during separation to free knuckle liquid. The influence of the milling and malaxation on the rheology of the slurry, as well as the separation process, are investigated.

Experimental characterization of olive oil pomace

After milling the olives, the malaxation process forms a homogeneous slurry with the oil, water and pomace being freed. Separation is reproduced in a lab centrifuge for 50 ml tubes, and planned in a lab scale decanter with rapid prototyping screws which are produced based on resin with a novel screw design to free knuckle liquid. Tests show that the milling and malaxation steps influence the rheological properties. The determination of viscosity, wall friction, ring shear rate, particle distribution, and moisture content was performed and used to gain data for the CFD and DEM simulation.

Discrete Element Method for solids transport

To enhance mixing of the pomace to allow olive oil to be set free, a novel screw geometry is developed using the Discrete Element Method. In comparison, while a traditional screw creates a highly homogeneous transport, the design with a changed geometry creates cake breaking and back mixing, which promises to free knuckle liquid and increase oil yield.

CFD simulation for flow optimization

The influence of the inlet position, additional discs and baffles is investigated by a CFD simulation in Ansys Fluent. Target is to avoid mixing of the already separated olive oil, and the best usage of the internal volume.

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LES-Euler/Lagrange Computations of Irregular-Shaped Particle Separation in a Hot-Gas Cyclone Based on a Random Transport Model

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Abstract

In biomass gasification plants, an important part of the process is the exhaust gas cleaning and specifically the separation of the dispersed particles consisting of ash, char and very fine aerosols. Due to the very wide particle size spectrum the separation unit consists of a cyclone and an additional fine particle separation device. In order to properly design the cyclone separator numerical simulations are conducted using a point-particle LES-Euler/Lagrange approach with two-way coupling. Since the particles in the exhaust gas are expectedly non-spherical in shape, the assumption of spherical particles will yield erroneous results regarding the separation efficiency. Therefore, in modelling particle transport and wall collisions, which frequently occur in a cyclone (Sommerfeld & Taborda, 2024), extensions are implemented accounting for an irregular shape of the particles. For this purpose, random processes are applied for the drag coefficient and the wall collision process as proposed by (Sommerfeld & Lain, 2018). Regarding the drag coefficient, the correlations proposed by (Laín et al., 2023) will be applied, which are depending on the sphericity of the particles. Additionally, the particle shape lift and the resulting torque is considered. The three-dimensional particle-wall collision model proposed by (Quintero et al., 2021) is based on a hard-sphere approach, but considers non-spherical particles in determining the contact point. Naturally, particle rotation has to be considered in the Lagrangian tracking.
For comparison also the (Haider & Levenspiel, 1989) stationary drag model incorporating sphericity (ϕ), will be applied and the results are compared with the stochastic model results.
The considered cyclone is operated under hot gas conditions, so that heat transfer between gas and particles will be considered through Nusselt number correlations, accounting for particle non-

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sphericity (Suri & Patel, 2024) . The results obtained with the non-spherical particle models will be compared among each other and also with the spherical particle models in order to assess the cyclone performance.

Acknowledgement

The present research is conducted within the Project Horizon Europe ResMe2E; Proposal number: 101172988. The financial support by the European Community is gratefully acknowledged.

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Innovative recycling of lithium-ion batteries using dielectrophoresis

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Abstract

Lithium-ion batteries (LIBs) remain the most common rechargeable battery type on the market. They are particularly relevant in the electric vehicle (EV) sector. Current LIB batteries for EV application have a life span of roughly 10 years and due to exponentially increasing EV sales, vast quantities of end-of-life batteries will accumulate in waste streams in the near future. Current recycling technologies, however, are energy intensive and cannot recover all materials, especially graphite.

Dielectrophoresis (DEP) is the movement of particles in inhomogeneous electric fields. It allows efficient and environmentally-friendly separation of microparticles based on their physical and chemical properties. DEP is mostly used in microfluidic separators for chemical or biomedical analysis. Recent developments, however, allow high-throughput separation that can address quantities several orders of magnitude larger than traditional DEP devices. One of those technologies is DEP filtration, the focus of this talk.

Dielectrophoresis is based on the action of an inhomogeneous electric field on a polarisable particle. Depending on the relative polarisability of the particle in the surrounding medium, particles move either towards local electric field maxima (positive DEP) or local electric field minima (negative DEP). The DEP movement velocity is directly dependent on the gradient of the electric field. To achieve the required field gradients, in DEP filtration, we scatter the electric field at the solid-liquid boundary of a porous material (filter) that is sandwiched between two sheet electrodes. Depending on the particle properties, particles will either be immobilised in the filter (positive DEP) or flow through the filter unaffected (weak DEP or negative DEP). This selective separation process is also switchable, as particles can be recovered by switching off the electric field (and thus switching off the particle holding forces). An important parameter that describes a particle’s relative polarisability (an important property in DEP) and thus the selectivity of the process is the particle conductivity. This

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dependence allows to separate a mixture of particles based on their conductivity value: conducting particles experience positive DEP, semi-conducting or non-conducting particles experience negative DEP.

In this talk, I will demonstrate how dielectrophoresis can be used as a pre-treatment for black mass, an intermediate product during battery recycling. Black mass is the fine fraction of shredded batteries and consists primarily of small particles of the cathode material (lithium-metal oxide, LMO) and the anode material (graphite).
The talk firstly shows how battery particles behave when experiencing dielectrophoresis, using pristine particles of the common battery materials NMC111, NMC611, NCA, and LFP, as well as spheroidized graphite. While graphite consistently shows positive DEP, most cathode particles display only weak or negative DEP. We attribute this to the much higher electrical conductivity of graphite compared to most cathode particles.

I will then show how this behaviour is leveraged to separate model black mass using DEP filtration at high throughput. We will show that graphite shows a significantly higher retention in the DEP filter compared to the cathode particles and we can rule out the particle size to be the deciding factor. We therefore attribute this to the diverging dielectrophoresis behaviour due to the difference in conductivity. We will also demonstrate how graphite can be enriched in a model black mass, a 50:50 mixture of pristine cathode material and graphite. Finally, we will address real black mass samples and demonstrate the relative enrichment of graphite in retentate and the relative enrichment of cathode particles in the filtrate.

DEP filtration is especially useful when dealing with particles that are 10 µm or below, because conventional techniques for material selective separation lose selectivity. Such a separation is not only useful as a preliminary sorting step in battery recycling but can be equally used for the recycling of other waste streams, e.g., to assist recycling of waste from electronic equipment.

PEM fuel cell recycling – A case study on mechanical separation via air classification

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Abstract

As fuel cell production numbers are predicted to rise significantly in the near future, fuel cell recycling is becoming an increasingly important issue. Mechanical separation processes like air classification can be used to separate polymer electrolyte membrane fuel cell (PEMFC) materials as the first step of an efficient recycling process chain. The quality of material separation is crucial for subsequent chemical and thermal recycling processes and affects achievable overall recycling rates.
In this study, we first outline strategies and possible process chains for efficient, holistic PEMFC recycling. We propose to divide recycling routes into (i) stack recycling, i.e. disassembly and separation of components or materials in order to obtain material fractions, and (ii) component recycling, i.e. individual processes for each material fraction in order to recover materials. In terms of stack recycling, PEMFC components can be separated from dismantled PEMFC stacks by automated disassembly, mechanical processes or a combination of both. Mechanical separation tends to be advantageous in terms of costs, especially for high throughputs, and adaptability to different stack designs.
For this case study, we initially focus on classifying and sorting shredded PEMFCs. We describe the mechanical material properties and discuss which materials of a fuel cell should theoretically be separable by air classification. We then present corresponding experimental results obtained with a zigzag sifter. Finally, we evaluate the results of the mechanical separation processes in the context of the entire PEMFC recycling process, deriving implications for previous and subsequent processes.

Content

EIT Based Visualization of Gas-Liquid Flow Patterns in a Horizontal Pipe

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Abstract

Identifying the flow regimes of gas-liquid mixtures in pipelines is crucial for monitoring and optimizing a wide range of industrial processes, especially those in the chemical, pharmaceutical, energy production, and oil-and-gas sectors. Flow regimes significantly influence the efficiency of processes involving mixing, separation, and transfer phenomena. To analyze these regimes effectively, tomography provides a powerful tool, enabling the reconstruction of phase distributions within the pipeline cross-section without interfering with the flow itself. Among tomography techniques, Electrical Impedance Tomography (EIT) stands out as a radiation-free technique, that requires a simple, inexpensive and safe hardware. In this study, the possibility of using EIT images of the flow to distinguish between different flow regimes in gas/liquid systems is explored. The EIT system used is composed of 32 electrodes, excited and measured using the adjacent current pattern. A 520 µS/cm water-NaCl solution was circulated in a 100 mm round pipe, with flow rate ranging from 0 m3/h up to 55 m3/h. A 5 mm injector was used to inject air at the center of the tube at 50 l/min, achieving stratified, plug/slug and bubbly flow regimes. Also, different surface tensions of the solution were tested, from 60 mN/m to 30 mN/m, to assess the influence of surface tension on the transition between flow patterns. The aim of this research is to demonstrate the feasibility of using EIT to distinguish between different flow regimes in gas/liquid flows, paving the way for implementing EIT in monitoring systems, particularly in processes where traditional techniques are impractical. Future works will focus on validating the method on more complex flow conditions, adding a third solid phase to the mixture.

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Particle sizing of polydisperse nano-suspensions using a broadband elastic light scattering (BELS) setup

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Abstract

The optimization of particle properties plays a decisive role in industry and research, for example in pharmaceutics or chemical science. Here, the focus is often on the particle shape and the particle size distribution (PSD). During the last years, particles in the nano-range gained increasing interest, e.g., in the release of active pharmaceutical ingredients. To produce particles with tailored properties, liquid phase processes such as emulsification are often used. To determine the PSD and to investigate the influence of process parameters on the PSD, online measurement techniques are crucial. Additionally, they are beneficial to control the size in continuous operations. Here, Elastic Light Scattering (ELS) is a very promising technique to measure the PSD of sub-micron sized particles in flowing liquids.
In this work, a new mobile setup based on Broadband Elastic Light Scattering (BELS) is presented. Instead of using multiple detection angles, this technique is based on the detection of continuous scattering spectra within a wide spectral range from the UV to the NIR, yet, at a fixed detection angle in backwards configuration. Therefore, the requirements regarding optical accessibility are drastically reduced for this inline measurement technique. We present the principle of the technique and the setup developed and show results from measurements on monodisperse reference particle samples as well as on particles with polydisperse PSDs. Different methods for the BELS-data evaluation are presented. In addition, the possibility of signal correction and the effect of multiple scattering for samples at higher concentration will be addressed.
The work was supported by the German Research Foundation (DFG) – project number 504809428.

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High-Resolution Cameras, Enhanced Data Transfer and Processing Techniques in Dynamic Image Analysis

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1. Introduction

Dynamic Image Analysis (DIA) has been an established and standardized method for determining particle size and shape for more than two decades [1, 2]. Like static methods such as microscopy, the quality and quantity of the images to be evaluated are of great importance in determining the result. While the image quality is a key factor in the evaluation of each single particle, a high number of particle images is essential for stable and reliable statistical results for both particle size and shape distributions.

A new commercially available camera with up to 20 MPix, 360 frames per second (fps) and data transfer via two channel CoaXPress (CXP-12) with maximum rates of 25 Gbit s^{-1} is successfully operated and used for DIA. Together with the flexibility to select image sections or levels of data compression, the camera is opening up a host of new possibilities for even more accurate determination of particle size and shape distributions.

An increased resolution with reduced noise even enables a new quality of determination of perimeter-based shape descriptors. However, the potential for more accurate evaluation can only be exploited if the grey image segmentation methods can keep pace with the data rate generated. In addition, it is particularly important to either avoid or reliably detect any blurring of the image to be able to make adequate and yet traceable corrections to the results.

The recent development of advanced and fast segmentation algorithms is therefore key to leveraging high-resolution particle images. Edge sharpness is assigned to each particle image as a quality characteristic for later decision making and can be displayed accordingly.

2. Fast image segmentation using an adaptive threshold algorithm

To exploit the features of the new camera, a fast algorithm based on text recognition is used to binarize the captured images. The algorithm uses an adaptive threshold in terms of a fixed percentage on the grey levels of neighbourhood pixels, what is more sensitive towards blurred particles compared to simple global or regional threshold values. Most valuable, the algorithm enables binarization rates (depending on the computer used) much higher than the highest frame rate of the camera. Therefore, it is possible to binarize each image more than once, what is used for image segmentation.

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In a first step, the whole image is binarized making a low percentage specification for the adaptive threshold to detect and locate all potential particles. The locations of these particles are used to define regions of interest (ROI) in the grey image. This way, additional information like the image sharpness of the particle can be extracted, which are normally lost after the binarization of the image. Fig. 1 shows the result of the segmentation of a SiC reference material. ROI are indicated with black boxes.

In the last step, the additional information is used to determine metadata like sharpness, decide whether the particle size and shape can be determined reliably or not and the particle can be included or must be excluded from further evaluation of the size and shape distribution. If necessary, the grey image ROI are re-binarized with a different threshold value specification.

3. Image sharpness of particles

The grey image segmentation enables the determination of particle metadata. In this work, the challenges and benefits of particle metadata are demonstrated by the definition of the (image) sharpness of the particle projection because it has a huge impact on the resulting binarized particle used for further calculation of size and shape parameters. Generally, blurred particles can contribute wrong values to the statistics.

Unfortunately, due to the essential requirement of well dispersed and not overlapping particles, the depth of the particle flow can extend to a couple of millimetres (especially for dry dispersion). Consequently, capturing particles out of focus cannot be avoided. Therefore, the identification of blurred particles is key to make adequate and traceable corrections to the result.

A major challenge is to define the sharpness of a particle in such a way that it can be determined independently of the particle size and even for particles as small as one pixel,

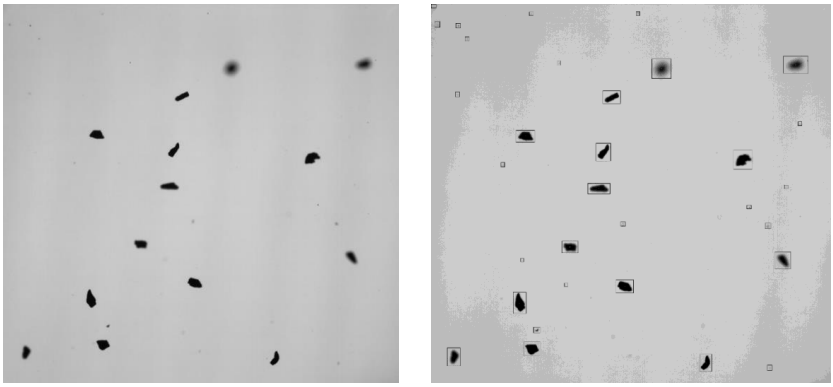

Fig. 1: Captured image of SiC particles (left) and corresponding segmentation (right).

otherwise particle number counting statistics are getting affected. Furthermore, it must allow to set a limit value to decide whether a particle can be included or must be excluded from calculating the size and shape parameters. Thereby, it is desirable that the limit value should be valid independently of the particle size, too. Different approaches exist to evaluate the sharpness of an image. Among them, statistically based focus measures such as the local variance method, the entropy of an image and Laplacian gradient [3] are of special interest because the computational effort to calculate them are rather small compared to more elaborated edge detector filters such as Prewitt or Sobel masks. First, a simulation is performed to evaluate the different focus measures mentioned above. Therefore, an image of a well-defined calibration reticle (40 µm Chromium dots on a glass slide, see Fig. 2) is captured and gradually blurred using a Gaussian filter. All images are segmented and the diameter of a circle of equal projection area (EQPC) and the focus measures are calculated. In the next step, the position of the reticle is changed in small steps of 0.04 mm. At each position, an image is captured and calculated accordingly.

From all the focus measures tested, only a few are suited to define the sharpness of a particle. Fig. 2 and Fig. 3 (left) show the result of the simulation for particle sizes of 40 µm.
As expected, the gradual blurring decreases the EQPC of the particles leading to wrong results. Noteworthy, a strong and unambiguous correlation between the EQPC and defined sharpness is evident, what is a basic requirement for a reliable sharpness measure.
For the simulation and the experiment, the EQPC remains nearly constant down to a normalized sharpness value of approximately 0.15, which would be an appropriate limit value, see Fig. 3 (left and right). Small deviations between the simulation and experiment are likely because small damages and light contamination on the surface of the reticle are getting focused when changing the reticle position, thereby disturbing the size calculation of particles.

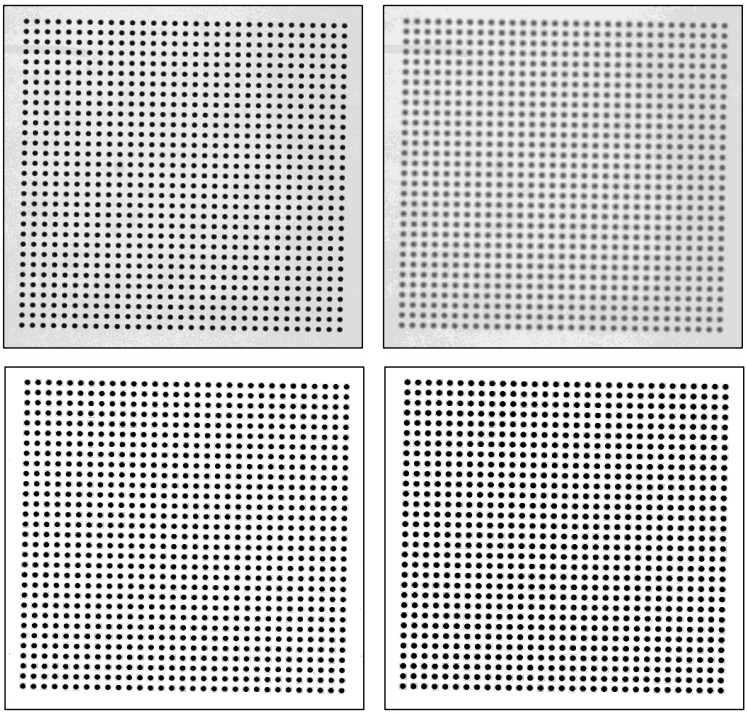

Fig. 2: Images of the calibrated reticle. Top: original image in focus (left) and blurred image using a Gaussian filter (right). Bottom: corresponding binarized images.

4. Conclusion

A camera with a resolution of up to 20 MPix and 360 frames per second is successfully used for DIA. An elaborated binarization algorithm enables to exploit these camera features and is used for image segmentation. The segmentation of the grey image allows to evaluate information of the particles, which are usually lost after binarization. In this work, it is demonstrated that the definition of an image sharpness of the particle is possible using a focus measure. This is particularly important to reliably detect blurred particles to make adequate and traceable corrections of the particle size and shape parameters. In future, an adaptive way to impact the thresholding in dependence of the sharpness of the particle is highly interesting.

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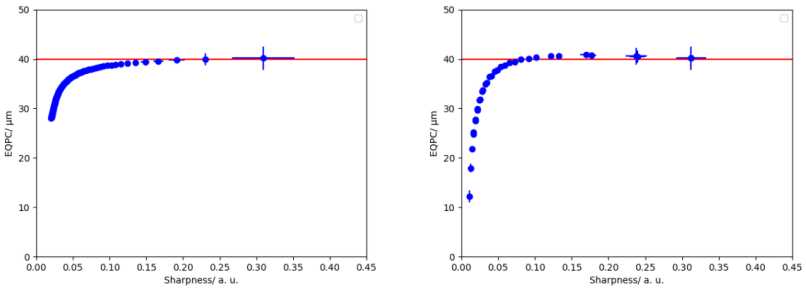

Fig. 3: EQPC for gradually blurred images (left) and for a calibrated reticle in different positions to the focus plane (right) in dependence of the sharpness.

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A novel 4-in-1 airflow cytometer for real-time analysis of particles

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Abstract

We present an innovative analysis system that combines several physical measurement methods and enables the high-resolution characterization of aerosol particles in real time. The SwisensPoleno Jupiter combines scattered light measurement, digital inline holography imaging, fluorescence spectroscopy (incl. lifetime measurement) and polarization analysis with powerful AI-based algorithms for automated data analysis.
The airflow cytometer relies on single particle measurement in the airflow and detects particles from 0.5 to 300 µm. For particles larger than 5 µm, holographic imaging with 0.6 µm pixel resolution is performed, with an error of less than 10% in size for 10 µm particles and larger. Smaller particles are characterized with light scattering measurements. In addition to morphological and optical characteristics, the autofluorescence properties of particles are analyzed, giving insights about their composition.
The real-time capability allows the automatic analysis of thousands of particles in complex aerosol mixtures and dynamic processes, such as particle atomization under variable conditions (e.g. temperature, humidity). A unique feature is the simultaneous analysis of morphology and chemical properties, which was previously not available in this form.
The data analysis is assisted by AI-based classification software, allowing application-specific classification for material testing or industrial quality control.
This system offers automated, precise particle characterization and opens up new perspectives for industrial applications where time-consuming manual methods were previously required.
Initial results of studies on the counting efficiency of round 10, 20 and 26um particles and pollen up to 100um show an average counting efficiency of 40%.

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Enhancing Powder Technology with Hybrid Physics-Inspired and Data-Driven Modeling

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Abstract

This study presents a hybrid modeling approach that combines physics-based insights with advanced data-driven techniques to enhance powder technology processes. Our methodology integrates deep-learning neural networks and graph attention networks with a genetic algorithm to effectively characterize and predict complex physical interactions within powder systems. This model excels beyond conventional simulations by providing a detailed analysis of material properties and their influence on process parameters, thus enabling precise control over manufacturing outcomes.

At the heart of our approach is the formulation of accurate symbolic representations that describe the complex relationships between process variables and system performance. The neural networks deals with unknown physical properties, while the genetic algorithm refines the generation and evaluation of symbolic expressions, ensuring greater precision.

The result of our research is a sophisticated 'grey-box' model that not only forecasts outcomes but also informs and enhances optimization strategies for powder processing. This presentation will detail our model’s development and illustrate its application through a case study, highlighting significant improvements in process efficiency. Our findings open new pathways for innovation and operational excellence in the powder industry, leveraging intelligent, physics-grounded modeling techniques.

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A Data-Driven Process Modelling Toolbox for an Enhanced Materials Production Understanding – Grinding Use case

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Abstract

The grinding process is a fundamental part of production in various heavy industries, from minerals processing to battery material. Optimizing its efficiency is crucial, as even minor improvements can lead to significant cost reductions and enhanced product quality. However, traditional methods such as trial and error or simulation, while offering acceptable results, have notable limitations, including high costs, time inefficiency, and the difficulty of accurately modeling complex interactions within the process.
To address these challenges a data-driven modeling pipeline was developed to enhance process optimization. This pipeline integrates advanced numerical and image data analysis, symbolic regression, and machine learning-based target optimization with a data limitation recommendation system, to provide a more empirical and systematic approach to process improvement. To ensure ease of adoption and future usability, a low-code toolset was created, allowing users to apply data-driven modeling without extensive programming expertise.
To showcase the effectiveness of this toolbox it was applied to the grinding process as a use case. Experimental and simulation-generated data were used to demonstrate how the pipeline enhances process understanding, providing deeper insights and more effective optimization strategies.

Content

Machine Learning Methods for Intelligent Digital Twins of Fluidized Bed Spray Granulation

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Abstract

Despite recent progress in automation technologies, the development of digital twins is still resource-intensive and slow as it requires large data sets, high domain expertise and reliable methods for the cyber-physical fusion. Our contribution aims to accelerate this complex development procedure particularly for fluidized bed spray granulation (FBSG). We therefore present various machine learning methods, which greatly contribute to the three core components of intelligent digital twins: Soft sensing, open-loop forecasting and multivariate control. We demonstrate a ML-based soft sensor architecture, that can infer the particle size distribution and particle water content from the most commonly available inline measurements in FBSG. The predictions from the soft sensor are used for real-time quality control and to warm up a subsequent forecasting model for these particle properties. This forecasting model consists of a recurrent neural network which is trained to generate an open-loop prediction of the particle size distribution and particle water content based on the desired path of the FBSG setpoints through time. The forecasts of various scenarios for different setpoint paths are then fed into a multivariate optimization algorithm to derive an optimized control decision within user-defined process constraints. The performance of these ML methods and their integration in the digital twin framework [1] are presented for different sets of experiments.

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Content

Submerged Plasma Synthesis of Graphitic Nanoparticles

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Abstract

Recent advancements in plasma technology have opened novel pathways for synthesizing carbon nanopowders with tailored properties. Here, we synthesize and compare graphitic particles produced by two types of plasma reactors.
First, a non-thermal dielectric barrier discharge (DBD) plasma is used to generate reactive species under non-equilibrium conditions. The plasma reactor is comprised of two electrodes separated by at least one dielectric and is powered by a sinusoidal voltage (1-20 kV). We synthesize carbon-based nanoparticles through the chemical decomposition of hydrocarbon gases (e.g., acetylene or ethylene) flowing through a helium discharge directed into a distilled water bath. These plasmas have drawn significant interest because of the lower operational costs, non-thermal nature, and reduced environmental impact.
We also investigate utilizing a thermal inductively-coupled plasma (ICP) reactor to produce graphene nanoflakes. In this process, a hydrocarbon precursor gas is introduced to the plasma, where the high temperature drives the rapid decomposition of molecules, with the products directed into a distilled water bath. High quenching and nucleation on microbubbles seem to be the mechanism that forms graphene nanoflakes.
The plasma conditions (such as power and gas flow rates) are parametrically studied for each case. Characterization of the nanoparticles is conducted and then compared to evaluate their quality and structural features, with correlation to the processing conditions. These findings highlight the potential of integrating both DBD and ICP into modern production lines, offering new paths for innovation in nanoparticle technology.

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Resynthesis of layered oxide cathode active materials from recycled batteries by coprecipitation

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Fraunhofer Institute for Surface Engineering and Thin Films IST

Abstract

Lithiated layered oxides such as Li[NixCoyMnz]O2 (NCM) play a fundamental role in the current efforts to electrify the transport sector. They are widely applied as cathode active material (CAM) in high energy density lithium ion batteries (LIBs) in order to provide battery electric vehicles with comparatively long driving ranges. Characteristically, lithiated layered oxides contain critical metals such as lithium, nickel and cobalt, the sourcing of which exerts geopolitical issues as well as a significant environmental impact. This issue can be addressed by establishing a circular economy for cathode active material, providing an alternative supply of critical metals for CAM production. In this context, current research efforts are being made to identify occurring impurities in recycled metal sources (e.g. nickel sulfate) and to evaluate their influence on the properties of resynthesized CAM. This study aimed to contribute to this research with the synthesis of layered oxide CAM (e.g. nickel-rich NCM) from recycled and primary metal salt educts in order to investigate their influence on the synthesis process and the resulting product properties. The applied process chain comprised a hydroxide coprecipitation process in order to synthesize a precursor (pCAM), followed by a lithiation and calcination step to convert the pCAM to CAM. The produced pCAM and CAM were characterized regarding their chemical composition (ICP-OES, EDX), morphology (REM) and particle size distribution (laser diffraction). Furthermore, the synthesized CAM was analyzed concerning its crystal structure (XRD) as well as its electrochemical performance within a coin cell.

Content

Interface-enhanced performance of noble-metal-free perovskite electrocatalysts for alkaline water electrolysis with mechanistic insights

Blaz Toplak[1]; Leon Müller[1]; Ali Raza Khan[2]; André Olean-Oliveira[2]; Philipp Gerschel[3]; Prof. Dr. Hartmut Wiggers[1]; Dr. Viktor Čolić[2]; Prof. Ulf-Peter Apfel[3]; Dr. Mohaned Hammad[1]; Prof. Dr.-Ing. Doris Segets[1]
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Abstract

The rapid depletion of conventional energy sources highlights the urgency for accelerated development of hydrogen technology. A key focus is developing efficient electrocatalysts from earth-abundant elements that can reduce overpotential in alkaline conditions. In this regard, perovskite materials emerge as promising, non-noble metal candidates for electrocatalysis. This research emphasizes a comprehensive material and electrode evaluation from synthesis to real-life testing conditions, with a particular focus on enhanced OER performance linked to the substrate/catalyst interface. Our approach not only demonstrated the high efficiency of perovskites for OER but also provided a better understanding of catalytic mechanisms.

In-depth electrochemical testing for OER under laboratory and industry-relevant conditions reveals that LaFeO3 consistently outperformed LaCoO3 and LaMnO3 in all the electrochemical measurements. To support these results, substrate/catalyst interface effects were investigated on glassy-carbon and nickel support. Furthermore, post-mortem analyses were performed to gain knowledge of the reaction mechanisms. Results revealed significant changes in material behavior under relevant electrochemical conditions connected to surface oxidation and amorphization of LaCoO3.

In conclusion, our findings emphasize the importance of optimizing the substrate/catalyst interface properties, which can significantly affect electrocatalyst performance due to improved electron and charge transfer. Deviating from the expected behavior, our comprehensive analysis provides a significant step towards understanding and developing highly efficient and stable OER electrocatalysts prepared from non-toxic elements.

Modelling microwave-assisted fluidized bed drying through DEM-CFD

Dr. Francesca Orsola Alfano PhD; Francesca Lucia; Francesco Paolo Di Maio; Prof. Alberto Di Renzo
University of Calabria (UNICAL)

Abstract

Industrial drying processes are essential across various sectors, including the treatment of waste ceramic materials. Fluidized bed drying offers significant advantages, such as high rates of moisture removal due to excellent gas-particle contact, resulting in efficient heat and mass transfer rates. On the other hand, microwave drying enables rapid and uniform heating, leading to shorter processing times and improved product quality. A combination of the two techniques can merge their benefits and overcome individual limitations, which is particularly important with low-added-value products, where maximizing drying efficiency is crucial to minimize costs. However, this field remains underexplored, necessitating experimental data obtained from complex laboratory equipment, yielding results that are challenging to interpret. Modeling tools can then serve as valuable allies in this context.
This study presents the development and implementation of a sophisticated modeling approach for simulating microwave-assisted fluidized bed dryers within DEM-CFD. The dielectric heating model integrates key phenomena, including microwave penetration depth into the fluidized bed, particle cohesiveness due to moisture content, progressive water evaporation, and the resultant changes in particle properties. Preliminary validation against microwave heating experiments confirms the accuracy of the model. The model makes it possible to carefully evaluate process feasibility, estimate power requirements, and identify potential product non-uniformities. The comprehensive approach highlights the potential of combining high-frequency electromagnetic waves with fluidized beds to optimize drying efficiency, reduce energy costs, and address industrial challenges.

Content

Improved bag filter operation with engineered particle properties and the challenges of correlating industrial data to lab scale tests

Dr. Rodney Foo; Dr. Johan Heiszwolf
Lhoist

Abstract

Industrial flue gases are commonly treated with bag filters to remove particulate matter which consist of process dust and air pollution control residues (APCr) when a dry flue gas treatment system is installed, The dust cake formation on the bag filters is necessary for high filtration efficiency (>99%) as the cake itself acts as filter with tortuous pores to trap particles as them move through the filter cake. However, the filter cake contributes significantly to the pressure drop as described by Dennis and Klemm therefore also the operating costs of the bag filter:

$\Delta P=(P\_E)\_\Delta W+CK\_2 V^2 \Delta t$

The constant, K2 for specific dust resistance is strongly driven by the cake structure and is a complex combination of many factors relating to the particle properties.

In this paper, we will present the benefits of engineering the right particle properties to reduce differential pressures over a bag filter in commercial applications. The challenges of characterizing and correlating lab scale powder characterization to real world industrial use will be discussed in detail.

Currently, K2 must be determined from specialist lab measurements, however, a limitation is that the actual filtration conditions typically cannot be adequately simulated experimentally. Therefore, semi-empirical correlations combined with industrial know-how are used to estimate K2 ranges for standard types of particles. Such an approach is however not sufficiently accurate when applied to new materials such as engineered specialty powder sorbents.

We will present the results of a novel and simple approach that links lab scale powder measurements and industrial bag filter differential pressure trends. This approach can be used to design particle properties that optimize the filtration in bag filters.

Content

Nanoparticle Design for Energy, Environmental and Health Applications

Prof. Dr. Sanjay Mathur; Prof. Dr. Sanjay Mathur
University of Cologne

Abstract

In the dynamic landscape of materials science, nanoparticle design remains a pivotal frontier to enhance the functional performance of specific materials and also to enhance the materials economy for target applications. Nanostructured ceramics and pigments play a key role as advanced photon harvesters, electrode materials in batteries and as electrocatalysts in energy conversion processes. In addition, conjugation of functional groups on the surface of nanoparticles enables immobilization of biocatalysts such as enzymes to degrade microplastics thereby improving the human environment. Moreover, development of biocompatible nanocarriers that can transport anti-tumor drugs in the body represent a major step in the future of precision medicine. For any systemically administered drug, the transport to the site of interest is inhibited by various physiological barriers, which reduces or even blocks the therapeutic efficiency of molecular drugs. Therefore, advanced drug-delivery systems are needed to overcome biological barriers. In this context, hollow silica nanoparticles functionalized with receptor-targeting ligands are promising drug-carriers to transport higher amounts of therapeutic payloads and to reduce any undesired off-site effects. Moreover, hollow nanoparticles can incorporate more than one drug enabling theranostic and theraregenerative approaches.

Charge Generation from Shower Heads

Prof. Dr. Tatsushi Matsuyama; Reo Nakano; Junich Ida
Soka University Japan

Abstract

The charge generation of liquid jets has been primarily studied in the context of industrial safety, particularly concerning oil and other flammable liquids. These studies aim to understand and mitigate electrostatic hazards. Additionally, charge generation in pure water has been of interest in the semiconductor industry, where it is commonly used as a cleaning liquid. Such liquids, characterized by their low conductivity, typically exhibit significant charge generation. In contrast, normal tap water, being more conductive, generates less charge and has attracted less research attention.
Showerheads generate water jets, and recently, new models incorporating advanced functions, such as ultra-fine bubble generators, have gained popularity (particularly in the Japanese market). There is growing interest in understanding how these new features influence charge generation in water jets.
To measure the current generated by a showerhead, a new experimental setup was designed. Current generation was evaluated for two newly marketed showerheads, a standard showerhead, and a setup where water flowed directly from a tube without a showerhead. The charge-to-mass ratio was significantly lower than the typical order of magnitude for charge generation (of powder). Interestingly, the charge generated by a standard showerhead was relatively higher than that of the other cases. In most cases, the generated charge was positive; however, when water flowed directly from a tube without a showerhead, it exhibited a negative charge. Notably, the charge-to-mass ratio was not significantly affected by variations in flow rate or pressure loss.

Electrostatic separation of nanoplastics from wastewater

Prof. Nicolas Vogel
Friedrich-Alexander-Universität Erlangen-Nürnberg

Abstract

Microplastics, generated by the degradation of plastic waste, have been found in nearly all ecosystems around the globe and continue to raise concerns as a widespread pollutant. Such micron-sized particles, however, may only be the tip of the iceberg. With sizes much smaller than 1µm, nanoplastic particles may be even more environmentally problematic as they can be taken up and interfere with individual cells of organisms. However, their small size makes identification and removal much more challenging compared to conventional microplastics. We aim to remove Nano plastic materials from wastewater by taking advantage of their colloidal properties. We synergetically combine electrophoretic deposition and particle-stabilized foams as techniques to remove such particles from wastewater.
We use model systems to understand the physical principles governing the separation and subsequently transfer the process to industrial wastewater and water-based paint as real environmental systems. The recovery percentage reached up to 98% and 95% from the model system and the industrial wastewater systems. Using a rotating electrode system, the process was successfully transferred to a continuous operation.

Content

Modeling the Complex Dynamic of Fine Grinding in Stirred Mills: A Unified Solid-Liquid Framework

Corlbert Ayuk; Dr. Olumide Ogunmodimu PhD
The Pennsylvania State University

Abstract

Fine grinding of slurry in stirred mills is a critical process in various industries, involving complex interactions between macro particles (rocks) and a fluid medium. As macro particles undergo mechanical deformation due to shearing forces and inter-particle collisions, fine particles are produced. These fine particles disperse in the liquid phase, altering its rheological properties and significantly increasing viscosity. This phenomenon introduces challenges such as increasing resistance to flow and potential inefficiencies in the grinding process. This study aims to address these challenges by developing a unified modeling framework that captures the coupled dynamics of the particles and fluid phases during fine grinding. Using a Lagrangian-Euler framework to model flow dynamics, and predict breakage probability function. The framework further incorporates the evolution of liquid-phase rheology in response to fine particle concentration and accounts for heat transfer generation through attrition, enabling an exploration of energy transport and the risk of thermal runaway. Our model offers insights into the intricate flow phenomena of stirred mill processes, providing a robust tool for optimizing grinding efficiency and ensuring operational stability. This work lays the foundation for enhancing the cost-effectiveness and sustainability of fine grinding operations.

The impact mechanisms of grinding aid additives on dry grinding

Tarek Sulaiman; Anderson Chagas; Prof. Dr. Arno Kwade PhD
TU Braunschweig

Abstract

Grinding aid additives (GA) are employed for dry grinding in media mills to enhance the overall efficiency of a grinding plant. Their mode of action is attributed to the reduction of the attractive forces between the product particles and to the improvement of the powder flowability. However, this improvement is accompanied with modifications in the powder bulk properties, which results in different process characteristics, such as the material hold-up and residence time within the grinding chamber, and subsequently in different product sizes. In order to account for these changes in the powder properties and the corresponding impact on the process, this study examined the relationships between important particles' characteristics, like the particle size and the specific surface area, as well as measurable bulk properties of the powder that effectively demonstrate the influence of GAs at different product sizes. For this purpose, calcium carbonate was used as test material, and three kinds of GAs were utilized. The results showed a correlation between the specific surface area measured with the BET technique and the flowability index ffc obtained from measurements with Schulz ring shear tester. The measured data could be fitted with power functions, which can dynamically predict the impact of various grinding aids on the powder flowability within the progress of the grinding process. Moreover, the data illustrated three grinding aid's efficiency areas. Utilizing the established correlation between the specific surface area and the powder flowability index, along an understanding of the optimal flowability index range for stable operation of tumbling ball mills, can enhance the predictability of grinding results with different GAs.

Content

Parameter Study and Optimization of Crusher Performance Using GPU-Accelerated DEM with Resolved Particle Fracture

Dr. Johannes Quist PhD[1]; Dr. Vedad Tojaga PhD[1]; Dr. Pontus Malmsköld[1]; Prof. Dr. Magnus Evertsson PhD[2]; Prof. Fredrik Edelvik PhD[1]
[1] Fraunhofer-Chalmers Centre; [2] Chalmers University of Technology

Abstract

Particle simulation methods that incorporate rock fracture are increasingly used in industry, yet their high computational demands have limited widespread adoption. In this work, we present a GPU-based dilated polyhedral discrete element method integrated with a bonded-element cohesive zone fracture model. A comprehensive parameter study on a cone crusher investigates the influence of key operating parameters—crusher speed, eccentric throw, closed side setting (CSS), and concave chamber geometry—on crusher performance. We further demonstrate how this DEM model can support performance optimization, showing its potential for future automated optimization workflows.

To this end, we introduce a parallel asynchronous optimization framework using Radial Basis Functions, with a Symmetric Latin Hypercube Design for the initial sampling. This framework highlights not only the value of efficient individual simulations, but also the benefits of leveraging a computational cluster in an automated, cost-effective manner. The parameter study results align well with established cone crusher performance behavior, and the optimization approach reveals different optima under varying rock material and feed conditions.

Content

A Benchmark Study on Wet Fluidization using CFD-DEM: Effects of Restitution Coefficient

Nicola Vanzetto; Prof. Dr. Niels Deen; Dr. Yali Tang
Eindhoven University of Technology

Abstract

Fluidized bed reactors are widely used in the chemical, pharmaceutical, and food industries. In these processes, particle-particle interactions play a crucial role in the reactor performance and, ultimately the product quality. With the presence of liquid in the reactor, inter-particle interactions are fundamentally different from that in dry conditions. A fundamental understanding of the micro-mechanisms of wet particle interactions and their macroscale impact is essential for a reliable prediction and precise control of the particle size evolution. For this reason, this study aims to advance the state-of-the-art towards a better understanding of wet agglomeration.

To achieve this, an Euler-Lagrange approach, commonly referred to as Computational Fluid Dynamics-Discrete Element Method (CFD-DEM), will be employed. One of the key advantages of CFD-DEM is its ability to incorporate a wide variety of inter-particle forces, which makes it particularly effective for studying the behavior of cohesive powders. In addition, DNS-derived closure models will be employed for the description of effective particle-fluid interactions, e.g. the drag force.

In such an unresolved Euler-Lagrange model, accurately representing particle-particle interactions is essential. The current abstract presents a benchmark study comparing gas-fluidization in dry and wet systems using CFD-DEM. The focus is on the effective description of wet particle-particle interaction using different approaches involving normal restitution coefficient, tangential restitution coefficient, friction coefficient, and liquid bridge force closure models. Ultimately, we aim to identify the most efficient and cost-effective method for wet fluidization modelling, validated by well-defined experiments.

Content

CFD-DEM simulation of cake formation and compaction in a centrifuge with an elastic-plastic model

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[1] University of Kaiserslautern-Landau (RPTU)

Abstract

Compressible cakes are commonly formed during filtration or centrifugation processes where particles settle and accumulate on a surface building up a cake over time. The applied fluid centrifugal pressure and forces can cause rearrangement, deformation or fracture of the particles, resulting in compaction of the particle packing.
In this study, the formation and compaction of a cake of elastic-plastic particles during centrifugation was analysed using experiments and simulations with a coupling of Computational Fluid Dynamics (CFD) and Discrete Element Method (DEM). Spherical elastic-plastic particles were suspended in ethanol and subjected to centrifugation in an analytical photocentrifuge. To investigate the plastic deformation of the particles depending on the acting centrifugal force, the rotational speed was increased and the temperature varied while the sediment height inside the centrifuge was measured. After centrifugation, the packing structure of the compacted sediments were analysed using µCT imaging. The CFD-DEM simulations were performed with a multiphase solver in which the centrifugal force, the Euler force and the Coriolis force were implemented. In the DEM, a previously developed elastic-plastic contact model [1] was used.
The packing structure of the cake, the acting forces and the plastic deformation of the particles were obtained from the CFD-DEM simulation. Based on the experimental and numerical data, the formation and compaction of the cake during centrifugation was described.
[1] Hesse, R., Lösch, P., Antonyuk, S.: CFD-DEM analysis of internal packing structure and pressure characteristics in compressible filter cakes using a novel elastic-plastic contact model, Advanced Powder Technology 34 (2023), https://doi.org/10.1016/j.apt.2023.104062.

On the Process and Property Function of Dry Water

Leigh Duncan Hamilton; Dr.-Ing. Harald Zetzener; Prof. Dr. Arno Kwade PhD
Technische Universität Braunschweig

Abstract

Dry water first appeared in 1964 when a patent was filed with the title: "Predominantly aqueous compositions in a fluffy powdery form approximating powdered solids". Since then, the high specific surface area of dry water has led to multiple applications, such as a carrier material for creams and powders, extinguishing fires or as a reaction catalyst. Dry water consists of microscale water droplets surrounded by a network of hydrophobic silica nanoparticles, whereby shear-driven mixing processes are mainly applied to form dry water. Even though dry water can contain up to 98 wt.% water, it appears as a powder with initially cohesive bulk behaviour. The cohesive properties can be overcome by slight agitation, resulting in a free-flowing powder.

This study investigates process as well as property functions of dry water. Firstly, the process is characterised for two mixing devices with an adapted stress model originating from grinding processes. The proposed stress model, composed of the stress intensity and stress number, enables predictions on energy-efficient parameter settings for a certain droplet size. The resulting, apparent powder properties are analysed using a ring shear tester and powder rheometer.

Initial results show optimal parameter settings using the stress intensity, whereas the hypothesised stress number seems decisive for the resulting droplet size. Additionally, dry water has unconventional bulk material properties. For instance, higher stress conditions in the ring shear tester altered the bulk material or the capsule condition, thus releasing water in the shear plane. Consequently, conclusions can be drawn about the mechanical stability of the sensitive capsules whilst providing interrelations between disperse properties and material handling.

Content

Tuning the spacing between inorganic nanoparticle cores in functional particle-based materials

Prof. Tobias Kraus
INM – Leibniz Institute for New Materials

Abstract

Inorganic nanoparticles lend materials and coatings strength, color, chemical reactivity and other desirable properties. Coupled nanoparticles enable electrical and thermal transport. The spacing between the inorganic cores affects material properties. In this talk, I will discuss what sets the spacing between inorganic nanoparticle cores with organic shells and outline routes to an improved control of the spacing.
In situ studies of nanoparticle agglomeration with optical and Small-Angle X-ray Scattering provide data on the spacing between nanoparticles as a function of core size and geometry, ligand shell structure, and solvent. I will discuss the relations between core-core attraction and shell-shell repulsion and introduce the core- and shell-dominated regimes of apolar nanoparticle agglomeration. A combination of experimental results and Molecular Dynamics simulations is shown to rationalize the spacings as a balance between core and shell interactions.
In the second part of my talk, I will discuss how the tunable spacing can be used to create “sensor materials” where nanoparticle cores react to stimuli such that the response can be used for sensing. As an outlook, I will discuss “reversible materials” where the spacing between inorganic cores has been tuned to enable function and dispersibility towards a new class of circular functional materials.

Content

Rationalisation of the interaction interplay among silica nanoparticles at surfactant-laden water-oil interfaces

Prof. Dr.-Ing. Lucio Colombi Ciacchi; Dr. Saeed Amiri; Dr. Michael Maas
University of Bremen

Abstract

Particle-stabilized emulsions are widely used in diverse areas such as oil recovery or water remediation, nutritional products, cosmetics and pharmaceutical formulations as well as in materials processing of composites and ceramics. Particles at the oil-water interfaces of emulsion droplets self-assemble in a rich portfolio of film morphologies, from gas-like phases through liquid-like chain assemblies all the way to condensed two-dimensional solids. Ideally, a control of such morphologies via adjustments of the emulsion formulation would be highly desirable. However, this requires a thorough understanding of the interaction interplay among the particles, the water-oil interfaces, the added surfactants as well as the temperature, pH, and ionic strength of the emulsion.

We have combined synchrotron X-ray reflectometry and dynamic interfacial tensiometry experiments will all-atom molecular dynamics simulations to unveil the fundamental nature of the interactions among APTES-coated silica particles at water-decane interfaces laden with octadecylamine surfactants. In particular, we have revealed the limits of applicability of mean-field DLVO theories to predict the local distribution of surfactant molecules around charged particles. Our results explain the previously postulated formation of an exclusion zone depleted from surfactants around each particle and its dependence on the ionic strength and the surfactant concentration. We also reveal a driving force for expulsions of particles away from the interface at a critical surfactant concentration, due to the build-up of in-plane stress upon increasing ordering of surfactant molecules.

This knowledge empowers us to develop more effective and stable emulsions for industrial and biomedical purposes.

Content

Measuring the stickiness of powders in relation to their temperature and moisture content

Dr. Gabrie M.H. Meesters; Jan de Jonge; Marise Eigeman
TU Delft

Abstract

Measuring the stickiness of powders in relation to their temperature and moisture content is of significant importance in the production of powders, esp. from liquids (Sewalt et al, KONA, 2021, DOI: 10.14356/kona.2021017). Specifically in spray dryers, fluid bed and drum dryers, where heat is used to remove the water. Also in coating related processes, the stickiness gives valuable information on spray rates of the coating solution in relation to the maximum temperature and humidity in the coat that can be set to prevent the particles from sticking to each other. Stickyness (Ts) or the glass transition (Tg) temperature are often reported and there is quite some variation in the both, which will be addressed in this presentation. Also, some known (DVS, DSC, Anton Paar, etc) and some novel techniques (e.g. Granudrum) will be discussed and the limitations of these techniques will be given, with respect to measuring the Ts and Tg. Also the rate of moisture uptake can be assessed by some of these techniques which is useful in packaging and dosing operations.

Content

Single Step Multiphase Fluid-Solid Coupling for Simulation of Wetted Particulate Systems

Dr.-Ing Stefan Endres PhD[1]; Prof. Dr. Lutz Mädler PhD[2]
[1] Leibniz Institute for Materials Engineering – IWT;
[2] University of Bremen / Leibniz-Institute for Materials Engineering – IWT

Abstract

Wetted particulate systems -such as humid aerosols, fluidised bed reactors, as well as imbibition, and drying processes of agglomerating particulate systems- present a particularly challenging problem for direct numerical simulation due to the need for 4-way coupled solid-liquid-vapour interactions. One way to alleviate the problem and to improve tractability is by deriving single time step ODE solutions for the combined fluid-solid systems which is possible using a discrete differential geometric (DDG) reformulation of the Cauchy momentum balance equation. Such simulations are essential for providing a deeper scientific understanding of the agglomeration mechanism, especially at small scales without dynamic experimental validation, in turn leading to improved engineering techniques. Additionally, because of the lack of experimental validation beyond measurable particle distributions, equilibrium state agglomerates and macroscopic properties it is essential that the simulation methods are able to display mesh independence. Mesh independence for a surface tension model was previously developed and the current work expands on such methods to finite volume method (FVM) integrators.

Content

Generative adversarial networks and stochastic modeling for reconstructing 3D particle morphologies from 2D images

Dr. Orkun Furat; Lukas Fuchs; Prof. Dr. Volker Schmidt
Ulm University

Abstract

Understanding the size, shape and inner structure of particles has become increasingly crucial for studying their structure-property relationships, e.g., the influence of particle descriptors on their processing behavior. Microscopic imaging techniques, like micro computed tomography, which can provide informative 3D image data of particles, are often not readily available. However, 3D image data is highly valuable not only for computing structural descriptors that quantify the particles but also for serving as input for spatially resolved simulations, enabling a deeper understanding of particle properties. Conversely, 2D image data acquisition, e.g., by scanning electron microscopy, is more feasible but it has limitations, e.g., quantifying 3D particles from 2D data is often non-trivial. This talk introduces a computational method to address these challenges by generating digital twins of 3D particles using stochastic 3D models calibrated with 2D image data [1]. The method employs parametric stochastic 3D models to generate random virtual 3D particles, consisting of particle shell and inner polycrystalline structure. Calibration of model parameters to 2D data is achieved through methods of generative artificial intelligence (AI). Specifically, a neural network is trained to guide selection of model parameter such that 2D cross-sections of generated particles statistically match the experimentally measured 2D image data.

References

[1] L. Fuchs, O. Furat, D.P. Finegan, J. Allen, F.L.E. Usseglio-Viretta, B. Ozdogru, P.J. Weddle, K. Smith, V. Schmidt. “Generating multi-scale Li-ion cathode particles with radial grain architectures, using stereological generative adversarial networks.” Communications Materials (in print).

MORe than DPM: Model Order Reduction for the Discrete Particle Method

Timo Plath[1]; Prof. Anthony Thornton PhD[1]; Dr. Stephan Rave[2]; Dr. Thomas Weinhart[1]
[1] University of Twente; [2] University of Munster

Abstract

Granular materials are pivotal in various industries, driving the need for digital twins to replicate industrial processes. However, their complex behaviours challenge traditional simulations. While the discrete particle method (DPM) accurately captures the key physics by solving Newton's laws per particle, its computational expense limits its application to large-scale scenarios. To overcome this, we propose integrating advanced open-source frameworks to enhance DPM efficiency through model order reduction (MOR).
Our approach reduces the complexity of DPM simulations by approximating particle behaviour using lower-dimensional models. This enables the development of efficient MOR methods tailored to granular material simulations, facilitating virtual prototyping and digital twin applications in industry.
Applying MOR to particle methods is challenging due to the random, high-dimensional dynamics of granular systems, which undermine traditional low-rank structures in state-space solution manifolds. To address this, we leverage MercuryCG: a specialised homogenisation tool developed by our team. This tool extracts continuum fields from particle data while conserving local mass and momentum.
Integrating this approach with MOR techniques mitigates the stochastic nature of particle simulations, enabling the creation of physics-informed surrogate models that retain the structure of the original DPM.
Our method bridges the gap between computational efficiency and physical accuracy, advancing the simulation of granular materials. By combining expertise and innovative tools, we introduce a transformative approach that offers unprecedented speed and reliability for process optimisation and design in industrial applications.

Content

Development of luminescent metal-organic frameworks particles as ratiometric thermometers

Lilong Wu; Christoph Huber; Dr. Dorothea Wisser; Dr. Florian M. Wisser; Prof. Dr. Martin Hartmann
Friedrich-Alexander-Universität Erlangen-Nürnberg

Abstract

In the field of chemical sensing, metal-organic frameworks (MOFs) have attracted much attention because they combine the advantages of high surface area and pore volume, crystallinity with tunable structures and functionalities. Thus, MOFs have found applications in easy-to-read optical sensors as well as in electrochemical sensing and fluorescence sensing. MOF-based fluorescence sensors are distinguished from other materials by their modular synthesis, which allows tuning the structure and chemical composition of the MOF and thus its optical properties. To enable ratiometric sensing, i.e. self-calibrating sensing without the use of an external reference, at least two different emissive centers are required. Here we present a series of mixed europium(Eu)-terbium(Tb) MOF particles together with a detailed investigation of their structural and morphological properties, including physisorption experiments, (variable temperature) powder X-ray diffraction and SEM. In addition, we will show how advanced solid-state NMR spectroscopy can be used to confirm the homogeneous incorporation of both cations in such dual emissive materials. Precise control of the Eu-to-Tb stoichiometry and distribution allows to control the luminescence properties of the final material. While the Tb emission is completely quenched at Eu loadings above 25 mol%, materials with lower Eu content enable ratiometric temperature sensing between 173 and 473 K. Interestingly, the emission profiles of the two luminescent centers show different behavior in the presence of organic solvents. We will discuss how, this solvation dependence of the Eu and Tb emission can be rationalized and taken into account in sensors, to determine temperature and discriminate between different solvents.

A novel approach for microplastic particles detection and differentiation

Prof. Dr. Pedro Faia[1]; Dr. Solange Magalhães[2]; Dr. Luís Alves[2]; Prof. Dr. Rasteiro Maria[2]
[1] University of Coimbra; [2] CERES, Department of Chemical Engineering, University of Coimbra, 3030 790 Coimbra, Portugal

Abstract

Plastic consumption has grown exponentially in the last decades, which has led to one of the most serious environmental contamination issues. Microplastics, MPs, result mainly from the fragmentation and degradation of large plastic materials. For many years, scientists have been trying to deal with the complexity of MPs detection and quantification, however, the lack of universal and validated methods led to a broad range of analytical approaches which are quite laborious, conducted off-line, and involving several steps and expensive equipment. Typically, they require intensive sample preparation, such as cleaning, drying and separation steps, that can potentially damage the MPs, and influence their analysis, and, mainly, do not allow continuous monitoring of the MPs. Electrical Impedance Spectroscopy, EIS, allows the analysis of a system's electrical response, yielding helpful information about its domain-dependent physical-chemical properties. Due to the superficial electronegativity of MPs' particles, EIS may be used to detect their presence, since the MP particles will change the overall charge and charge transportation paths in the suspensions containing them. In the present work an on-site fast and reliable EIS based system was developed, with the capacity to detect different types of MPs (Polyethylene, PE, Polyethylene Terephthalate, PET, and Polyvinyl Chloride, PVC), as well estimate their concentration. The developed strategy has proved, so far, to be capable not only of differentiating suspensions containing particles of the mentioned types and variable concentrations (in the range between 0.001 to 0.166 g/ml, with accuracies above 97%), as well as from mixtures containing simultaneously different plastic types.

Content

Particle Fingerprint: Reveiling Material Composition in Large-Scale Particle-Discrete Datasets

Dr. Ralf Ditscherlein; Dr. Thomas Buchwald;
Prof. Dr.-Ing. Urs A. Peuker
TU Bergakademie Freiberg

Abstract

The Particle Fingerprint introduces an innovative visualization technique for efficiently analyzing large-scale particle-discrete data. This approach translates gray value histograms into stacked, sorted two-dimensional patterns, encoding frequency data as grayscale for intuitive visual interpretation. By utilizing X-ray tomography particle-discrete datasets, this method captures compositional variations across thousands of particles while preserving essential distribution characteristics. The Particle Fingerprint is particularly valuable for quality control, process monitoring, and the analysis of complex particle systems. This study outlines the methodology, including image preprocessing, gray value normalization, and histogram stacking, and demonstrates its application using Python. Results from synthetic and real datasets highlight the potential of this visualization to reveal underlying patterns and compositional trends, even in highly complex systems. Future applications could include dynamic process monitoring and batch marking, establishing the Particle Fingerprint as a powerful tool for particle system analysis.

Content

Genetic Algorithms in Ultrasonic Extinction for Particle Size Analysis

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Sympatec GmbH, Clausthal-Zellerfeld, Germany

1. Introduction

Whenever light-optical methods for particle characterisation require a considerable amount of preparation work, due to the opacity of the dispersion, ultrasonic extinction measurements offer efficient solutions for the determination of particle size distribution (PSD) and concentration. The Sympatec OPUS system analyses highly concentrated suspensions between 5 and 50% by volume without affecting its original state by dilution. It measures the frequency-dependent loss of ultrasonic intensity caused by the interaction of the particles with the sound wave. The knowledge of an extinction function is required for the calculation of the PSD from the measured data. Its calculation represents the major challenge, especially for unknown substances or mixtures. The software KSIGMA calculates the extinction coefficients of the solid matter of interest. The program has recently been improved by means of genetic algorithms to obtain optimal results within short calculation times for even unknown substances and mixtures.

2. Instrument

The ultrasonic extinction spectrometer OPUS analyses particle size distribution as well as concentration of the suspension or emulsion ranging from below 1 µm up to 3 mm (see Fig. 1). Instead of electromagnetic waves (light), ultrasonic extinction applies low energetic sound waves. These waves are passing through a suspension or emulsion with varying frequencies. For the introduction as well as the detection of sound waves, a piezo-electric RF-generator and detector set-up is used. The recorded loss of sound energy depends on the size and concentration of particles. A corresponding attenuation spectrum is determined and converted into a particle size distribution (PSD). Using sound instead of light waves, ultrasonic extinction is independent of the level of transparency. Hence, totally opaque suspensions like ore slurries, water in crude oil or coke slurries, can be analysed. Furthermore, paints, sugar pastes, or pharmaceutical suspensions with high solid contents, rank as standard applications for the ultrasonic extinction principle. In addition, the ultrasonic extinction method allows a rugged and robust probe design that resists typical crude process conditions, especially in mining industry.

Content

The OPUS probe masters temperatures up to 120°C, pressures up to 40 bar and the entire range of pH-value.[1]

Fig. 1: Ultrasonic extinction spectrometer OPUS

3. Software

KSIGMA is a software program developed by Sympatec (see Fig. 2) and used to calculate the projection-area-related extinction cross section, which describes the attenuation of sound energy by solid particles. Based on OPUS extinction measurements and corresponding particle size distributions the program returns the product specific extinction function. The corresponding particle size distributions must be determined by orthogonal measuring techniques such as laser diffraction (LD, with e.g. HELOS), dynamic image analysis (DIA, e.g. with QICPIC), sieving, or by manual input.

The extinction function is determined by using a task specific genetic algorithm according to the flow chart in Fig. 3. A variety of reliable measurement data is helpful for the calculation. This variety is provided by different size fractions of the product, each of which corresponds to exactly one PSD. The suspensions of the fractions may be available in different concentrations. These basic measurement data are easily loaded to the program and in addition to its main task of calculating the extinction function, KSIGMA offers the option of displaying the measurement data in various diagrams. These diagrams provide a quick visual comparison of the presented attenuation measurement data and enable an assessment of its quality and consistency.

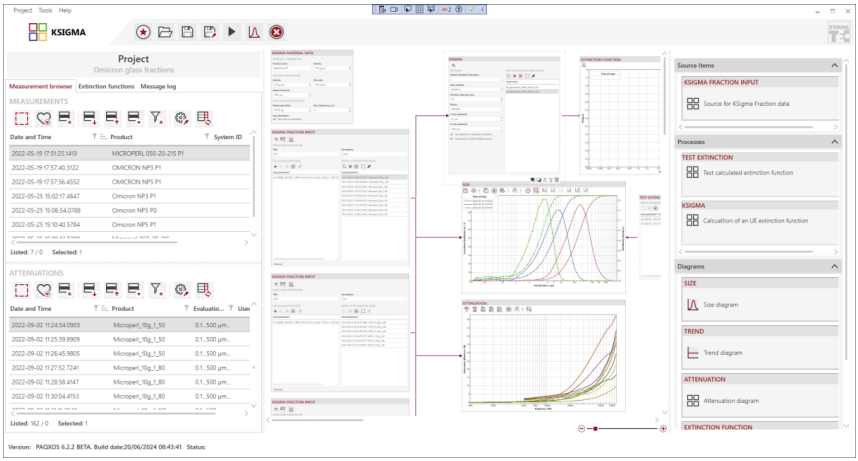

Fig. 2: Depiction of the KSIGMA software.

4. Process of the genetic algorithm

As depicted in Fig. 3, the algorithm starts with a variable amount of initial extinction functions. These functions are ranked and listed using a suitable and well-predefined fitness function. A fixed percentage of the fittest candidates (related to their specific fitness function) is selected.

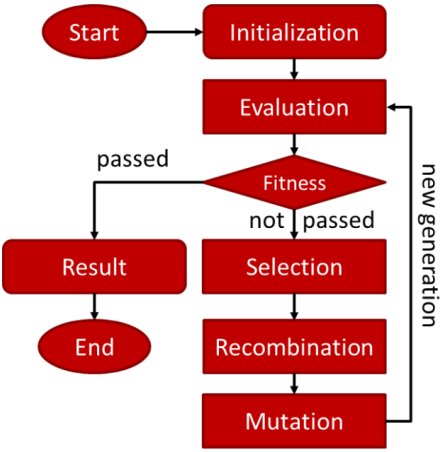

Fig. 3: Process scheme of the genetic algorithm used in KSIGMA.

In numerous iterations, successive changes are made to the initial extinction functions. The changes are achieved through recombination and individual parameters of the newly generated functions are modified by random mutation. They form the new generation and are re-evaluated. Once the algorithm's stopping criterion is reached, the result, the determined extinction function, is output.

5. Validation

The KSIGMA program outputs a slightly different result with each calculation, due to the nature of the algorithm to deal with random numbers. To qualify the output, a confidence interval of 95% for the silicon carbide (SiC) P600 has successfully been accomplished in each 100-fold repetition of calculation. Furthermore, a data set of four different size fractions of SiC (P50, P80, P600, F1200) has been added as specification to the KSIGMA program to calculate the size-independent extinction function for SiC.

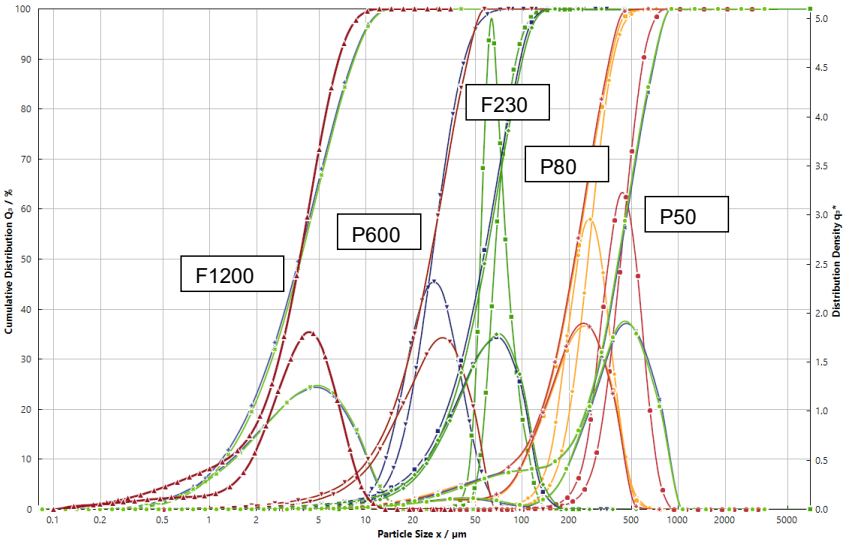

Fig. 4: Comparison of the input distributions (narrow) and the evaluated distributions (broader at equal $x_{50,3}$ sizes) from KSIGMA results for different fractions of SiC, obtained using the genetic algorithm. The fraction F230 was no used as input data for the algorithm. The result of F230 (in comparison to the (narrow) result from an alternative technique) shows the validation of the method.

As a functional proof for validation of the method, the F230 fraction of SiC, which was not part of the input calculation data, has been evaluated along with the other SiC fractions and all evaluations show matching results with the input calculation data concerning the $x_{50,3}$. For the fraction of F230 with no input calculation data the result is compared with the (narrow) result from an orthogonal technique instead. All calculated distributions (see Fig. 4) are slightly broader than the input data, due to the enormous range that the extinction function covers to fulfil all requirements. In total, the genetic algorithm, manages to generate an extinction function that suits all demands coming from the evaluation of OPUS extinction measurements.

6. Conclusion

Extinction coefficients can now be determined with genetic algorithms from reference results obtained for different size fractions and concentrations. The development of the new algorithm includes the variation of the iteration progress-dependent parameters, such as mutation probability, optimisation of recombination, and the definition of a suitable fitness function. The validation of the method or algorithm was shown by analysing size fractions of SiC material which were not part of the input data.

A general and effective form of a fitness function is developed and will be presented. It offers an option to easily unite several different self-defined aims to the function. The passing on of promising modifications will be presented as an accelerator for computation. The problem of local minima and how to avoid them will be discussed with respect to initial parameters and multicore threading.

The advantage of using the new genetic algorithms is that neither knowledge of all inevitable process-related influences, nor knowledge of all underlying processes that contribute to the extinction are necessary to obtain optimal results for the evaluation of the PSD.

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Green hydrogen via alkaline water splitting: the impact of microstructure on anode layer properties and performance

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Abstract

Alkaline water electrolysis and alkaline anion exchange membrane water electrolysis are crucial for the generation of green hydrogen. This work addresses the impact of anode layer microstructure on the oxygen evolution reaction (OER), examining why variations in Ni-Co-O thin film morphologies affect performance and how anode properties are intricately linked to the microstructure of the layers [1]. By fabricating particle-based anodes with similar mass loading and chemical characteristics, the effect of microstructure on mass transport at the three-phase contact and thus activity and stability during the electrochemical OER could be unraveled.

Key findings include the following microstructure-property-performance relations: In comparison to pure water, water-ethanol mixtures improve particle stability in the ink, enhancing particle connectivity within the layers and thereby reducing ionic and contact resistance. Higher drying temperatures produce smoother morphologies with reduced lyophobicity that can be analytically described by the Cassie-Baxter-model. In contrast, lower drying temperatures yield increased roughness. These enhance lyophobicity of the anodes, and thus increase the availability of active sites by promoting effective removal of bubbles from surface of the layers during OER.

The found correlations enable rational design of anode layers for the alkaline OER. They bridge from microstructure via anode layer properties to performance and thus provide the understanding that is essential for high-performance alkaline electrolysis applications.

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Solar Fuel Production by Thermochemical Particle Processes

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Abstract

The use of thermochemical cycles for splitting water with the help of concentrated solar power provides an alternative and sustainable route for hydrogen production. Together with the ability to simultaneously split CO_2 into carbon monoxide, these processes enable the production of synthesis gas, which is the building block for synthetic fuels and also widely used in the chemical industry. The splitting in a thermochemical two-step cycles uses a redox material to divide the process in a reduction and an oxidation step. During the reduction step (1), the redox material is regenerated by using high temperatures up to 1500 °C to release oxygen from its lattice structure. By exposing the activated material to water vapor and/or carbon dioxide at temperatures around 800-1000°C, oxygen ions are split off the gases and the material is re-oxidized, leaving hydrogen and/or carbon monoxide as product (2).

$$\mathrm{M_{ox}} \rightarrow \mathrm{M_{red}} + \mathrm{O_2} \quad (1)$$

$$\mathrm{M_{red}} + \mathrm{H_2O/CO_2} \rightarrow \mathrm{M_{ox}} + \mathrm{H_2/CO} \quad (2)$$

Several processes using this concept were developed in the past, using mainly locally fixed monolithic structures of the redox materials [1-4]. Their operation could show the general feasibility but suffered from inefficiency because of heat losses due to necessary temperature swings.

This work will present alternative concepts with solid, moving particles of the redox material, which overcome aforementioned restrictions and allow better scalability. The solid particles are moved in a closed cycle. They are heated directly under concentrated solar irradiation in a receiver to maximum temperature and transported to the subsequent process units. The reactions are separated spatially in different components, which allow for continuous operation. Particle/gas heat exchangers allow for heat recovery and modelling results suggest solar-to-fuel efficiencies up to 20% [5].

Content

Test settings and results of reactors operating with particles under concentrated irradiation will be presented. DEM/CFD simulations were used for design optimization of particle transport and flow. Furthermore, the particle selection and their properties will be addressed, as they influence the flow behavior and affect component design. Finally, current challenges regarding the material, design and operation will be discussed.

References:

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Sustainable Polymer Processing using Supercritical Fluids and Sustainable Additives in Particulate Form

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Abstract

The rising demand for environmentally friendly materials in engineering applications has prompted significant research into sustainable polymer processing techniques. This study explores the innovative use of supercritical fluid technology [1], in conjunction with eco-friendly additives in particulate form, such as rice husk ash and wollastonite [2], to enhance the properties of biodegradable polymers for advanced engineering applications.
Our research focuses on the hydrothermal pre-treatment and the subsequent supercritical foaming of polymers, including among others, polycaprolactone (PCL) and polyvinyl acetate (PVA), to develop porous scaffolds with unique properties [3]. We thoroughly characterised the additives measuring their particle size distribution, shape, and chemical composition to understand their influence on pore formation and structural integrity in the resulting scaffolds. The incorporation of these particulate additives aims to facilitate pore formation and improve structural integrity while maintaining environmental sustainability. Rigorous testing was conducted to establish optimal processing conditions, ensuring significant improvements in porosity and mechanical strength.
Preliminary results demonstrate that the addition of wollastonite and rice husk ash significantly enhances the performance of the scaffolds, offering a promising approach to developing materials that meet both sustainability goals and performance criteria in engineering applications.
The potential of supercritical fluid technology combined with natural particulate additives is pivotal in advancing sustainable material science, paving the way for innovative solutions in particle technology and broader engineering challenges.

Content

Mechanochemically assisted enzymatic processing of cotton textiles

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Abstract

The continuous growth of the fashion industry has resulted in a significant increase in textile waste generation. To address the challenges of used textiles, the European Union has mandated separate collection systems for these materials, starting in January 2025. This regulatory framework facilitates the management of significant waste streams and offers potential for developing innovative product pathways that can convert materials destined for incineration or landfills into valuable resources through advanced processing techniques and plant design.
The utilization of cotton from textile waste presents challenges due to its high degree of crystallinity and the complex nature of mandatory depolymerization steps. Additionally, contamination and variability in the composition of textile waste must be carefully considered. The application of enzymatic hydrolysis provides opportunities for conducting processes in a sustainable and selective manner. Consequently, a process is developed that integrates mechanical energy input with enzymatic treatment. A wet rotor mill is utilized to introduce the required mechanical energy into the water-textile suspension and to improve the accessibility of cotton.
This study investigates various combinations of milling and enzymatic degradation of cellulose fibers derived from used textiles. A key focus is on identifying optimal process parameters that can further enhance the process, thereby improving both efficiency and economic viability while ensuring high quality in the final product. An important parameter under consideration is the influence of energy input within the milling system. By establishing these optimal process parameters, this research contributes to the advancement of sustainable textile recycling technologies.

Content

Influence of Dispersion Gas on FexOy formed in Spray Flame Synthesis

Orlando Massopo
Paderborn University

Abstract

This study investigates the impact of atomization on the morphology, size distribution, and crystallinity of maghemite nanoparticles, as well as remaining impurities in the product utilizing standardized SpraySyn burners 1.0 and 2.0. The investigation specifically focuses on varying the dispersion gas flow rates (DG). Droplet dynamics and mean droplet size were directly measured in the spray flame using PDA, followed by an evaluation of its impact on particle formation. The inline size distribution of aggregates extracted via a Hole in a Tube system was assessed using a SMPS. Characterization of the product was performed through TGA-MS, TEM, XRD and Raman spectrometry. PDA measurements indicate an increase of the DG results in a reduction of the mean droplet size along the flame axis. This observation suggests an enhanced efficiency of the atomization and evaporation of the precursor solution at elevated DG, with the evaporation completion occurring after the pilot flame at the highest DG. A significant correlation exists between DG, spray flame morphology, and height, which subsequently affects the high-temperature particle residence time (HTPRT) within the spray flame. As nanoparticle growth is influenced by HTPRT, an increase of the DG directly impacts nanoparticle formation owing to the enhanced cooling effect associated with higher DG levels. TEM imaging demonstrates a reduction in primary nanoparticle size by approximately 50% as the DG increases from 6 to 12 slm, with nanoparticles exhibiting high homogeneity in shape and low polydispersity, indicating a predominant gas-to-particle conversion pathway. BET and XRD data support the TEM. TGA-MS measurements showed an increase in carbonaceous species with increasing DG, suggesting the synthesis of less-pure product.

Content

Flame-made Quantum Dots

Dr. Keroles Riad PhD
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Abstract

Quantum dots have unique size-dependent properties and promising applications. The applications of metal oxide quantum dots range from photocatalysis (TiO2), UV protection films (ZnO), gas sensing (SnO2), and high-temperature superconductors (CuO). Further, we have recently shown that semiconducting nanoparticles such as TiO2 and ZnO can photo-polymerize epoxy via cationic polymerization. However, the lack of viable quantum dot mass-production processes hinder their use in many applications. Quantum dots are usually synthesized using wet chemistry techniques that are relatively complex, require hours, and have a production rate in the order of grams per day. On the other hand, flame spray pyrolysis (FSP) is a one-step process that produces metal oxide nanoparticles at industrial rates in the order of tonnes per day. Here [1], we synthesize quantum dots of four metal oxides embedded in a silica matrix in a one-step mass-production process using flame spray pyrolysis.

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Influence of surface impurities on the catalytic activity of nanoparticles synthesized by a spray flame synthesis

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Prof. Dr. Ing. Hans-Joachim Schmid M.Sc.
Paderborn University

Abstract

The exhaust gas of combustion processes – particularly for biomass combustion – typically contains a significant amount of nitrogen oxides (NOx). The prevailing technology for exhaust gas denitrification is selective catalytic reduction (SCR), which involves the catalytically enabled conversion of harmful NOx with ammonia to the harmless species nitrogen and water. Commonly, this is done using vanadia/titania catalysts, which are e.g. processed in filter bags or installed as standalone catalyst modules.
An alternative SCR technology is currently being investigated and involves the addition of catalytically active nanoparticles to an exhaust gas stream. SCR occurs in-flight at the freshly created particle surface. Downstream, the catalyst particles are precipitated with the fly ash particles in the surface filter. Because the nanoparticles are still reactive, the filter cake serves as a fixed bed catalyst and therefore enhances the NOx separation performance. Experimental results with iron oxide and manganese oxide particles prove the efficacy of this novel SCR concept. However, some challenges remain. Contrary to what is often claimed, spray flame synthesis does not yield pure particles. They contain significant amounts of carbonaceous impurities. These impurities inhibit the adsorption as well as the activation of NH3 and NOx on active sites at the particle surface. The SCR performance in dependence of the influence of carbon residues and particle properties on SCR efficiency will be presented for this novel SCR°technique which is a promising approach and has the potential to become an attractive upgrade measure for exhaust gas cleaning systems with an existing precoat filtration stage.

Content

Pilot-scale spray-flame synthesis of iron oxide nanoparticles: Investigation a hydrogen-based burner concept

Martin Underberg
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Abstract

The production of nanoparticles in spray flames is a versatile method for the production of materials with different compositions, morphologies and properties. To proceed from lab-scale experiments to industrial applications, pilot-scale studies are an important intermediate step. In this study, we use a pilot-scale burner design based on an H2/air pilot flame stabilized by an electrically heated ring. By using H2 and the biofuel ethanol, the burner enables the sustainable production of nanoparticles. The temperature distribution in the flame profile is a critical parameter that influence the decomposition of the precursor as well as the nucleation and the growth of the particles. Therefore, we have performed spatially resolved measurements of the gas phase temperatures within the reactive zone using non-invasive NO-LIF multiline temperature measurements for different process conditions. These results were combined with measurements on the synthesis of iron oxide nanoparticles using a precursor solution of iron nitrate nonahydrate in ethanol. We varied the process conditions, including variations in fuel type, dispersion gas and flow rates and measured the resulting temperature distribution and, finally, correlated this with the product characteristics. The product particles were analyzed with electron microscopy for morphology and particle size distribution, BET gas adsorption to determine the specific surface area, and X-ray diffraction to identify crystallite phases and sizes. The combined methods enable the identification and explanation of correlations between the operating conditions, the gas phase temperature and the product properties. It will be shown that the dispersion gas flow rate influences the temperature-time profile but has only a minor effect.

Content

Defects in particles – a widely unknown territory

Prof. Dr.-Ing. Wolfgang Peukert PhD

Abstract

An important trend in particle technology is the requirement to understand and design systems of increasing complexity. The property function defines the target in product design and is understood along the five dimensions of size, shape, surface, composition and structure, and their respective distributions. The presentation will focus on defects in particles, the least understood and investigated dimension of the particle property space. Defects in single particles occur inside of a particle or at its surface. A major difficulty in studying defects is their in-depth characterization, which often requires sophisticated and expensive techniques including advanced spectroscopic and scattering techniques and electron microscopy.

In size reduction, the particle strength is controlled by defects. In particular, the grinding limit strongly depends on the ability of nanoparticles to store defects. In contrast, the delamination from bulk to form 2D materials (e.g. graphene or MoS2) must be tuned to minimize defect formation. In particle syntheses, tailoring the shape of anisotropic plasmonic particles such as gold requires careful control of defects in the seeds from which the desired particle shape evolves. Defects in semiconductors determine the band gap energy, while defects at a catalyst surface determine the catalytic conversion. Defects are also highly relevant for functional devices including solar cells, electrodes in batteries and fuel cells. Based on the given examples, we will discuss white fields to show unique opportunities for further research of fundamental questions and in pressing fields including energy transformation on the way towards “green” particle technology.

Content

Supraparticles for Enhanced Lithium-Ion Battery Anodes: Improved Processability and Electrochemical Performance of Si/C Materials via Hierarchical and Porous Structures

Dr. Fatih Özcan; Adil Amin; Moritz Loewenich; Lars Grebener; Dr. Mohaned Hammad; Simon Heckenbach; Mena-Alexander Kräenbring; Ahammed Suhail Odungat; Atharva Harshawardhan Ladole; Thai Binh Nguyen; Daniel Schwabenland; Hasan Kadah Salim; Prof. Dr. Hartmut
University of Duisburg-Essen

Abstract

The demand for eco-friendly energy solutions drives research on advanced materials for energy applications, while scalable manufacturing processes are key for industrial adoption. Nano-sized silicon-based anode materials, a promising alternative to graphite in lithium-ion batteries, attract attention due to their potential to enhance energy storage performance. Recent advancements include spray-drying techniques to create hierarchical electrode structures that address silicon's volume changes during cycling. These assemblies reduce active material exposure, minimize irreversible reactions during solid-electrolyte interphase (SEI) formation, and improve handling, processing, and electrode packing. However, their success depends heavily on the mechanical stability and size of the structured supraparticles.

This study examines the influence of supraparticle size on the properties and performance of Si/C composite anodes. Size distributions impact key factors such as interparticle spacing, slurry rheology, coating density, and electrochemical behavior. Supraparticles with a median size of 5.0 µm achieve the highest coating density at 0.90 g cm^{-3}, resulting in uniform layers with controlled thickness and porosity, yielding superior cycling stability. Smaller supraparticles, while offering good layer control, form less dense coatings with more SEI formation, reducing initial Coulombic efficiency. Optimizing supraparticle size distribution balances porosity, mechanical stability, and slurry performance, providing a pathway to improve battery performance and scalability for next-generation energy storage systems.

Content

When particles get wet – micro scale insights into particle collisions

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Abstract

Macro scale solid processes, such as mixing, pneumatic conveying or fluidization, consist of a variety of different micro processes or phenomena like agglomeration caused by interparticle collisions. Conversely, with the help of the individual micro processes that occur, it is possible to describe the macro processes. For the design and modelling of such macro processes or for product design, knowledge about these micro processes is necessary. In industrial operations like fluidized bed spray agglomeration, the solid bulk materials are often not dry, but are wetted by a liquid. This significantly increases the number of possible microscale phenomena in comparison with dry operations.
This work aims to provide a fundamental understanding of the micro phenomena caused by interparticle collisions under dry and wet conditions. To investigate the mechanisms, single particle experiments were performed by means of a newly developed setup, to realize binary collisions of two particles under dry and wet conditions. The collisions are recorded by two high-speed cameras, enabling motion detection in three-dimensional space by classical and AI-supported methods of digital image analysis. The positions, translational velocity, rotation and the amount of liquid on the particles are determined both before and after the collision. The results allow a direct comparison with DEM collision models, both with and without additional liquid. The results show the influence of system properties such as surface roughness and material pairing on the collision outcome and highlight the limitations of existing DEM models.
The authors would like to thank the German Research Foundation (DFG) for funding the project via HE 4526/28-3.

Green Chemistry in ball mills: Mechanical stress shaping reaction kinetics

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Abstract

Mechanochemical methods enable sustainable and efficient chemical reactions. Mechanical stressing activates the reactants and triggers chemical transformations without the need for solvents. Previous investigations in simple setups using a model reaction (1) have shown that both the stress type and stress intensity have a significant influence on the reaction kinetics.

$Na_2CO_3 + CaCl_2 \rightarrow CaCO_3 + 2\ NaCl$ (1)

These findings are now being transferred to grinding media mills, in which impact, compressive and shear stress occurs together. For the investigations, media mills with different movement patterns were selected (high-energy, planetary and mixer ball mill), whose grinding media introduce the mechanical energy into the reactants at different normal and tangential speeds. With the help of DEM simulations, the impact and shear influences can be analysed separately by quantification of the effective stress energies. Experiments in the respective mills allow a correlation of the stress conditions from the simulations with the experimentally determined reaction kinetics, whereby the stress type, number and intensity were in focus. It can be shown that the dominant stress mechanism varies as function of the mill and that a certain minimum stress intensity is beneficial for an efficient chemical conversion. During processing, significant caking of the powder on the grinding chamber and media was observed, which changes the amount of captured powder and the elasticity of the impacts. Both variables influence the stress intensity and, thus, potentially the reaction kinetics. By combining numerical and experimental data, new insights into the stress conditions of grinding media mills and their influence on the reaction kinetics of a mechanochemical model reaction were gained.

Content

Mechanistic modelling of multi-component comminution in stirred media mills

Maximilian Tobaben; Prof. Dr. Arno Kwade PhD
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Abstract

The comminution of multi-component systems is of great importance in many industries. Multi-component systems are ubiquitous, particularly in the field of minerals engineering. While the comminution of individual components in stirred media mills has already been intensively studied and optimised in the past, there is a lack of systematic research into the comminution of multi-component systems in the micrometre range. In particular, the interaction of the individual components, including different material properties and different capture behaviour by the grinding media, makes the precise control of multi-component comminution in stirred media mills complex.To address this challenge, this study focuses on the experimental investigation of the comminution of a binary material mixture of quartz and limestone and on the description of the comminution processes using the mechanistic stress model according to Kwade (2003). The aim of this approach is to illustrate the influence of the stress conditions on the selectivity, which can be regarded as a measure of the separability of two materials. The central aspect is the stress intensity. This is defined as the ratio of the stress energy to the mass of the stressed particles and takes into account both the main process parameters as well as the mill geometry and the particle size of the feed material. The results show a strong correlation between the stress intensity and the selectivity, while the influence of the mixture composition is negligible within the analysed range. In general, it can be seen that the stress model offers the possibility of determining suitable process parameters for effective and selective comminution with knowledge of the material hardnesses.

Content

Multicomponent breakage – a combined experimental and numerical approach

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TU Braunschweig, Institut für Partikeltechnik

Abstract

Understanding the breakage behavior of materials through systematic experiments and numeri-cal simulations is crucial for the design and optimization of comminution processes across vari-ous industries. Slags, as multicomponent systems rich in valuable minerals and metals, present challenges due to their heterogeneous structure and complex chemical composition. Detailed characterisation of their fracture properties as a function of structural properties and composition is essential for efficient recycling strategies, particularly in a circular economy.

This study investigates the breakage behavior of slag particles with varying compositions, focus-ing on fine particles and the transition from multicomponent to single-compound systems. Exper-imental investigations were conducted using a two-roller breakage tester, enabling controlled, reproducible analysis of forces, energies, and particle size distributions under variable pressure conditions. Complementing these experiments, a numerical simulation framework using the Dis-crete Element Method (DEM, Ansys Rocky) was developed to simulate particle breakage behav-ior, analyze mechanisms, and explore factors influencing specific energy consumption.

By integrating experimental and numerical approaches, breakage functions for multicomponent systems were developed and calibrated. These insights form the basis for optimized comminution processes and sustainable recycling strategies for complex materials like slag.

Content

Influence of different surfactants on the adhesion forces between oil-based binding liquids and minerals measured with a FluidFM® system: Correlation of selective wet agglomeration behaviour and agglomerate structure

Laura Schwan; Prof. Dr. Ulrich Bröckel

Abstract

This work investigates the influence of different surfactant types on the selective wet agglomeration process of valuable minerals from crushed slag phases (gangue). The adhesion forces between droplets of binding liquids and suspended model particles treated with different surfactants are measured using atomic force microscopy with microfluidics (FluidFM®). The results will be used to describe the agglomeration process in terms of selectivity. It will be determined whether greater differences in adhesion forces between the minerals and gangue types lead to improved selectivity. The influence of stirrer speed and thus shear forces on the agglomeration process will also be investigated, as well as the structure of the resulting agglomerates by using microtomographic images.

Content

Microstructure analysis and determination of the fracture mechanism of different slag types using X-ray computed tomography

Thu Trang Vo; Prof. Dr.-Ing. Urs A. Peuker
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Abstract

The liberation of valuable minerals from its ore requires comminution to get the valuable mineral exposed and is mainly influenced by the underlying fracture mechanisms. Fracture mechanisms have their boundary classification in non-random and random breakages. Random breakage does not show any dependency with the properties of the parent particle, e.g. mineralogical composition, shape and size of the valuable phase. Meanwhile, the non-random breakage would result in a breakage that is completely dependent on the properties of the parent particle and leads to the full exposure of the desired mineral phase.
In the priority program SPP 2315, slag is investigated as a carrier of valuable minerals that contain elements classified as critical by the EU commission. These elements are transferred from the molten metal phase to slag during the metallurgical process, where they crystallize as engineered artificial mineral (EnAM). The study focusses on characterizing slag which exhibit granular and dendritic structures of the lithium aluminate, identified as the valuable mineral, with its fracture behavior.
To determine the fracture mechanism, EnAMs and gangue material need to be characterized, e.g. the intergrowth within the gangue material. Slag samples with different structures are subjected to compression in a load cell. Fracturing is then captured by using X-Ray computed tomography, supported with light microscopy and SEM. Particular emphasis is placed on: The condition of unbroken slag samples, the degree of mineral liberation, the size distribution of fragments, the proportion of valuable mineral within the fragments and the morphology and progression of fracture surfaces.

Content

Leveraging Powder Characterisation to Predict Dissolution and Dispersion in Pharmaceutical Formulations

Khizra Abdul Wadood[1]; Dr. Christopher Windows-Yule PhD[1]; Dr. Matthew Molloy[2]; Dr. Andrew Ingram[1]
[1] University of Birmingham; [2] AstraZeneca

Abstract

The dissolution and dispersion of pharmaceutical powders are critical parameters influencing drug efficacy and manufacturing efficiency. Predicting these behaviours from fundamental powder properties is challenging, especially as lumps and aggregates often form during powder-solvent mixing, compromising product quality. This study addresses these issues by experimentally characterising pharmaceutical excipients to identify key material attributes governing dissolution performance.

Advanced techniques are used to conduct quantitative measurements of particle size distribution, flowability, and cohesion. These data offer insights into how particle-level attributes influence dissolution stages, including wetting, sinking, swelling, agglomeration, and dispersion. Systematic comparisons between powder properties and controlled dissolution experiments reveal which critical quality attributes dictate dissolution rate and extent.

Building on these findings, a data-driven computational framework is developed using deep learning algorithms. Training predictive models on high-quality characterisation and dissolution data reduce reliance on time-consuming experimental trials. It streamlines formulation development, accelerates process scale-up, and improves the reliability of predicting product performance under varied conditions.

The outcomes highlight the importance of linking material properties to dissolution mechanisms. This enables targeted formulation optimisation, consistent product quality, and enhanced scalability. More importantly, it ensures reliable prediction of dissolution profiles early in the design phase, supporting the development of safer, more effective pharmaceutical products.

Content

Insights into pneumatic conveying of a granular phase

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Mines Saint-Etienne

Abstract

Pneumatic conveying provides an extremely rich industrial context to study coupling interactions between granular and fluid phases. The variety of pneumatic conveying regimes ranges from dilute regimes, when the carrying fluid flow rate is large, to dense regimes, when the carrying fluid flow rate decreases. It requires various types of modelling to offer a better understanding of this complex interaction. The transport of solid particles by a flowing fluid phase relies on viscous drag. However this picture becomes rapidly much more complex if this viscous dissipation of the energy is associated with solid friction due to long-lasting contacts between particles, or between particles and confining walls.
Despite the relative simplicity of pneumatic conveying systems, where a fluid and a granular phases are mixed together at inlet of and then transported along a duct through a pressure drop, the variety of conveying regimes is often studied very empirically, for a particular type of granular material and conveying configuration. In the present work we propose to revisit this issue by stepping back towards more general description of conveying regimes. We will illustrate the strong analogies between pneumatic and hydraulic conveying, stemming from a common viscous drag mechanism of the granular phase. Through experimental data collected on a table-top and pilot-scale apparatuses, we will explore the phase diagram proposed by Kalman (2020). Eventually a slug statistical analysis is performed on a large number of slug displacements together with PIV giving insights into the stochastic dynamics and the granular flow associated with intermittent traveling structures in dense conveying regimes.

Content

Automated Process Optimization in Mixing with Adaptive and Dynamic Mixing Modes

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Abstract

Mixing plays a crucial role in industry, for e.g., fine and petrochemicals, food, pharmaceuticals, and mineral processing. The selection of the stirrer is based on the desired flow pattern and the rheological properties of the material being processed. A change in the mixing task or a significant shift in material properties during the process can lead to decreased efficiency, longer mixing times, or poorer mixing quality. To address the need for handling various tasks across a wide range of applications with the same mixer, a multi-purpose mixer with adjustable tools was developed. It consists of a coaxial stirrer with an inner and an outer stirrer. The blades of the inner stirrer can be changed in inclination during the process. This allows different tasks, such as dispersion, aeration, and suspension, to be performed efficiently in succession without the need for tool changes. Additionally, the process parameters can be dynamically changed to compensate for changes in the process. Energy input into the fluid serves as a key parameter to quantify the quality of a dispersion process. The three independent actuators can be used to achieve the best energy input. To identify the optimal settings, a control algorithm called the "automated design of experience" was developed.
This algorithm adjusts the speed of the main and coaxial stirrers and the blade angle in a sequential manner, evaluating whether these changes lead to improved or worsened energy input. It is hence possible to determine optimum mixing parameters in an automated way.

Content

Coupling of experiments and DEM simulation for powder flow understanding and prediction

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Abstract

The context of this study is the prediction of hopper discharge in nuclear fuel fabrication. This stage of the process is sensitive to the flow properties of the powder. The methodology presented here is based on the creation of a numerical model of the FT4 powder rheometer, which is commonly used to assess the flowability of powders. The considered FT4 geometry corresponds to a rotating blade passing through a powder bed (VFR test configuration).
Two approaches are utilized to achieve this objective. Firstly, the discrete element method (DEM), is used to calculate contact forces and deduce the velocity fields of powder particles. Secondly, an experimental approach is adopted, whereby particles are set in movement while acquiring images using a high-speed camera. Image analysis is used to study the movement and the velocity of the particles. These two methodologies are applied to the study of the behavior of millimeter-sized particles in the VFR geometry (Glass particles, whether spherical or non-spherical, and UO2 granules).
On one hand, calibration is carried out by comparing the numerical results with the experimental data, focusing on the force couple measured by the blade during VFR tests. On the other hand, particle imaging velocimetry (PIV) is utilized to characterize the particle movement around the blade. A comparison is made between the numerical and experimental results using the velocity fields and the thickness of the shear band.
This method allows a better understanding of the particle flow and the validation of the results of the numerical simulation through experimentations at laboratory scale. It enables us to consider the simulation of process operations involving substantial quantities of powders.

Content

Design Space and Control Strategy for the Manufacturing of Drug Nanocrystal Suspension by Wet Media Milling

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Abstract

Wet media milling is a fully industrialized technology for the manufacturing of drug nanocrystal suspension [1]. This work describes the development of an advanced control strategy and an associated design space for a manufacturing process at commercial scale. The development was facilitated by a comprehensive Quality-by-Design (QbD) approach to identify the relationship between material attributes, process parameters and quality attributes. The current status and challenges for the industrial application of QbD are described elsewhere [2, 3].

Full scale experiments and mechanistic process modeling have been used to establish a physical reasonable control strategy of factors relevant to the quality attributes of the nanocrystal suspension. The design space has been developed based on a mature mechanistic process model of the wet media milling procedure [4]. It represents the process-product quality attribute relationship between a multidimensional range of measured process parameters and the manufactured mean particle size of the drug nanocrystal suspension. The control strategy allows for simple, robust and sound scientific process control as well as operational flexibility of the suspension batch size [5]. This is an industrial case study of control strategy and design space definition with the crucial contribution of mechanistic process modeling for an intended commercial manufacturing process.

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Content

Advancing dynamic flowsheet simulation for complex solids processes with Dyssol

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Abstract

Dynamic modeling of interconnected solids processes is an important yet underrepresented topic in process engineering. Traditional approaches often fail to handle complex solid process dynamics due to the need to account for distributed parameters of granular material, such as particle size, porosity, or moisture content, which are critical for industrial applications. Dyssol [1], an open-source dynamic flowsheet modeling environment, was developed to address these challenges. It combines advanced algorithms and numerical methods to provide a solution for simulating granular solids and their dynamic interactions. The unique capabilities of Dyssol include support for both dynamic and steady-state models, multidimensional distributed solid properties, and an extensible library of unit operations models. To ensure maximum flexibility and applicability, the program applies the sequential-modular approach to calculations. This allows integrating new heterogeneous models of virtually any complexity, and using a variety of internal algorithms and numerical methods – from differential equations to data-driven techniques [2]. As a result, the simulation environment has already been shown to be applicable to a wide variety of processes, such as agglomeration, granulation, crystallization, drying, chemical looping combustion, battery recycling, minerals grinding, ceramic tiles [3] and zeolite production [2].

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Content

The Control strategy Evaluation Tool (CET): Theoretical Background

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Abstract

Characterization of pharmaceutical processes is generally restricted to a small design space. High-fidelity digital twins allow for exploring a larger design space and running numerous experiments virtually. This project aimed to develop a systematic approach to build digital twin of continuous manufacturing lines based on advanced discrete element model (DEM) simulations. The digital twin relies on simulating typical pharmaceutical materials, equipment, and processes using verified numerical methods and algorithms. Using this platform, different quality control strategies can be designed and evaluated to perform corrective actions.

Using a continuous direct compression line as an example, a DEM model is employed to simulate powder flow and predict the residence time distribution (RTD) in a horizontal blender connected to a three-paddled feed frame. The RTD from the DEM simulations is then used to parametrize and interpolate reduced-order models (ROM) to predict the system response at various process settings. The collected RTD information, including DEM data, ROMs, and ROM interpolation algorithms, is a core component for a Control Strategy Evaluation Tool (CET).The CET offers the possibility of building digital twins of continuous manufacturing lines and provides a systematic framework for implementing quality control strategies The CET can be used to assess the effect of various types of process disturbances and to evaluate different quality control strategies. Additionally, the CET offers the possibility to include experimental data, compare CET predictions to process analytical technology tools, and compare the respective discharge actions.

Disclaimer: This abstract reflects the views of the authors and should not be constructed to represent FDA’s views or policie.

Content

Influence of droplet size distribution on coating homogeneity in hot melt coating

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Abstract

Central quality requirement in hot melt coating processes is creating a homogeneous coating across one particle (intra-particle homogeneity) and in the entire batch (batch homogeneity). Both depend on the droplet size distribution of the coating material. However, the droplet size distribution usually is unknown and achieving a high coating quality necessitates high experimental effort.
Our new test setup using laser diffraction enables the measurement of droplet size distributions of the coating material directly as introduced in the fluidized bed. By varying the atomization parameters, their effect on the resulting droplet size distribution can be investigated. The measured droplet size distributions then serve as input parameters of a Monte-Carlo model. With this model a single particle can be coated based on the measured droplet size distribution in silico. During the coating process, random spots on the particle are covered with droplets resulting in a local coating thickness increase. During the process, however, droplets can encounter already coated areas, leading to an inhomogeneous distribution of coating material on the particle.
With the newly established experimental setup, the droplet size distribution of the coating material becomes a fundamental basis for further optimization of the coating process. The developed model offers the possibility to analyze the droplet size distribution in relation to the produced coated product quality. After the simulated process, the intra- and inter-particle coating homogeneity can be evaluated. Varying the input parameters allows the identification of the optimum set of process parameters for a given material combination and their transfer to the experimental setup.

Content

Characterizing the particle movement and deposition during the thermal runaway of lithium ion batteries in confined spaces

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Abstract

The thermal runaway (TR) presents a significant safety concern in applications using lithium ion batteries. During TR battery cells release a particle-gas-jet through a burst vent. In applications battery cells are housed in battery packs, where the vented gas-particle stream is confined within the casing, creating a significant risk of thermal propagation (TP). Existing studies primarily address gas dynamics and thermal effects, despite interactions with particles such as heat conduction, deposition and abrasion having a major influence in a TP scenario.

While crucial for safe battery design, the particle movement and deposition is as of yet poorly understood. In particular, the high pressure and temperature environment makes characterisation challenging. In this work, we present a novel experimental test setup designed to study particle dynamics under TR conditions in a confined battery like environment.

The experimental setup consists of a battery cell venting into a 20 mm high steel channel. Heat and pressure resistant viewports enable insitu imaging of the particle trajectories. Using image analysis methods we were able to estimate the massflow and demonstrating the particle ejection during TR occuring in transient pulses. In addition sedimented particles were retrieved along the channel length for laboratory analysis. Thus we were able to spatially resolve particle mass, particle composition and size distributions leading to novel insights into the deposition behavior . For further validation the experiments are aided with CFD-DEM Simulations of the particle deposition in the battery casing.

Our findings provide critical insights into gas-solid multiphase flow behavior in battery TR scenarios, contributing to the advancement of safer energy storage technologies.

Content

3D hydrodynamics of a draft tube spout-fluid bed: Insights from magnetic resonance velocimetry

Jens Patrick Metzger[1]; Boyuan Chen[2]; Prof. Dr. Alexander Penn[3]; Dr. Christian Guenthner[4]; Prof. Dr. Klaas P. Pruessmann[4]; Prof. Dr. Christoph Müller[1]
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Abstract

Draft tube spout-fluid beds (DTSFB) are common reactors in industrial applications such as particle drying and coating due to their enhanced heat and mass transfer between the gas and solid phase. The draft tube creates a distinct flow pattern, dividing the solid motion into a slow-moving annular region and a faster spouting region within the draft tube. The height of the draft tube gap plays a critical role in controlling both the rate of particle circulation and the duration of gas-solid contact.
However, the opaque nature of granular materials poses a significant challenge to visually analyze the internal hydrodynamics of DTSFBs. Hence, semicircular beds or visual probes are commonly employed, which inadvertently disrupt particle flow through the addition of extra walls or probes to the system.
This study uses magnetic resonance imaging (MRI) to non-invasively investigate the hydrodynamics of the solid phase in a 3D DTSFB. The flow of oil-filled particles in a lab-scale DTSFB is measured in a 3T MRI scanner under various operational conditions. Solid distribution maps and particle velocity maps are generated to assess the entrainment of solid material from the annular region to the draft tube. The results uncover flow phenomena such as a vena contracta flow at the draft tube entrance and the suppression of gas bubbling in the annular region. Complementary simulations using CFD-DEM confirm the experimental observations and augment the measurements with insights into the gas flow field.

Content

Laser meter of aerosol microparticles parameters during their spontaneous transformation

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Institute of Information Technologies and Systems of the National Academy of Science of Ukraine

Abstract

The presentation discusses a laser dual-wavelength microparticle analyzer that, unlike classical methods, allows for real-time observation of spontaneous transformation of water microparticles, for example, into H2O2 [1] and other aerosol transformations. The features of the spatial structure of the high-frequency signal and the method for calculating its main parameters for the scheme of a laser two-wave analyzer of microparticles with symmetrical reception of scattered radiation relative to the OYZ plane under different polarization states of the probing beams are also considered:

1) the beams have linear coordinated states of polarization with the direction of oscillation of the electric vectors perpendicular to the OYZ plane, or have linear polarizations with the direction of oscillation of the electric vectors in the OYZ plane;

2) the first probing beam has a right-handed (left-handed) polarization, and the second probing beam has a left-handed (right-handed) polarization.

Calculations of different schemes of a laser dual-wave microparticle analyzer are presented using a developed application program on a computer. Conclusions were drawn about the effectiveness of these schemes. The most efficient scheme of a laser dual-wave microparticle analyzer is proposed, namely a laser dual-wave microparticle analyzer with symmetrical reception of forward-scattered radiation.

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Ultrasonic Monitoring of Zeolite and MOF Crystallization

Prof. Dr. Martin Hartmann; Dr. Marcus Fischer; Rebecca Reber; Prof. Dr. Wilhelm Schwieger
Friedrich-Alexander-Universität Erlangen-Nürnberg (FAU)

Abstract

The investigation of the underlying mechanisms for the crystallization of molecular sieve materials such as metal-organic frameworks (MOFs) and zeolites have received increasing interest in recent years. This is mainly because improvements of the corresponding equipment were made that is essential for in situ diagnostics. In contrast to classical techniques, such as X-ray diffraction and neutron scattering, nuclear magnetic resonance or infrared and Raman spectroscopy, ultrasonic monitoring has not received proper attention. In the present study, we report the use of the ultrasonic device as a real-time, in- situ diagnostic tool for monitoring the crystallization progress of zeolite A from homogeneous, colloidal solutions. Kinetic information like reaction rate or reaction order of crystal growth can easily be calculated from the measured ultrasonic signal data. For the mathematical description of the crystallization curves, the experimental curves were fitted with different kinetic models. The understanding of metal organic framework syntheses is still a challenging task in the field of porous materials. Proposed models for the MOF formation mechanisms includes the formation of pre-nucleation building units (PNBU). The nature of these PNBUs is widely discussed, but detailed insights are still rare. In this study, ZIF-8 with varying particle size and morphology is synthesized from zinc nitrate hexahydrate, 2-methylimidazole and CTAB in different solvents. In our study, we found that the ultrasonic attenuation already increases rapidly within the first minute of the ZIF-8 syntheses. Depending on the molar ratio of linker to metal, not only the final particle size, but also the course of the ultrasonic signal changes.

Novel options for the prediction of solid oral dosage dissolution

Dr. Stephan Sacher; Dr. Andreas Kottlan; Dr. Elisabeth Fink; Prof. Johannes G. Khinast
Research Center Pharmaceutical Engineering GmbH

Abstract

With the implementation of advanced processing concepts such as continuous operation, pharmaceutical manufacturing is becoming more efficient. Especially, the time span from powder raw materials to the final solid product is significantly shorter. One remaining bottleneck in modern manufacturing is the release procedure, in which major critical quality attributes (CQAs) must be proven to be within the specified limits. The dissolution performance of a solid drug product is a crucial CQA as it defines the bioavailability of the drug in the human body. Conventional in-vitro dissolution testing is a tedious and time-consuming approach.
In this talk alternative methods for the prediction of the dissolution profile will be presented. The first approach utilizes a combination of gas in scattering media absorption spectroscopy (GASMAS) and photon time of flight spectroscopy (pTOFS) to measure the optical porosity of tablets. The optical porosity is correlated with the parameters of a dissolution model via a data-driven approach. The second presented approach is via optical coherence tomography (OCT). OCT typically measures the coating thickness of coated pellets or tablets, but also structure-related image features of tablet cores can be extracted. The coating thickness or other image features are again correlated with a dissolution model. For both approaches, the model parameters, which are predicted from in-line or at-line measurements, are used to compute the dissolution profile. All concepts have been demonstrated for pharmaceutical products from different manufacturing routes including continuous wet granulation, hot melt extrusion, pan coating and fluid bed coating. Both novel methods show promising results as enablers for real-time release testing (RTRT).

Content

Production of disperse systems by (melt-)emulsification

Leah Ebner; Dr. Jochen Schmidt
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Abstract

Emulsions are used in several fields of the food-, pharmaceutical and cosmetics industries. Their key properties, such as shelf-life, rheology and texture are defined by the droplet size distribution of the dispersed phase and long-term stability of the system. Understanding the dynamics of these processes is therefore crucial for optimising emulsification processes and adapting the properties of the resulting emulsion.
In melt emulsification the dispersed phase, which is a solid at ambient temperature, is emulsified at elevated temperatures in the molten state. Once the emulsion is cooled back to room temperature, the droplets solidify and spherical particles are obtained. This makes the system less dynamic and by freezing the system state, it is possible to observe the particle size distribution depending on e.g. the process time and the chosen stressing conditions. Due to this possibility, melt emulsification besides formulation of emulsion systems is also well suited for fundamental investigations of emulsification processes.
This contribution presents parameter studies on the melt emulsion model systems paraffin wax-water-Tween 85 and montan wax-water-SDS. Emulsification is performed in a melt-emulsification plant which consists of a stirred tank, where the pre-mixed liquid phases and emulsifier are pre-emulsified. Then, the pre-emulsion is fed by a gear pump to the rotor-stator unit , where droplet-breakup occurs under turbulent flow conditions. The product is circulated back to this stirred tank. The process is performed continuously for a period of 60 minutes at constant operating parameters. Sampling takes place before and directly after the rotor-stator unit. The samples obtained are immediately cooled in order to solidify the dispersed phase and, thus, to stabilise the dispersion in its actual state. The influences of emulsifier concentration, residence time in the stress zone, energy input and process time on the development of the particle size distribution during melt emulsification are observed.
The particle size distribution (PSD) is determined off-line using laser diffraction. In addition, SEM images of the products are taken to analyse their particle shape and structure. The

Content

dependence of the PSD on process parameters and system composition was studied systematically.

The results show that overall smaller droplet sizes can be achieved for paraffin wax as an oil phase than for montan wax. General prerequisites for obtaining small droplets are flows in the turbulent regime and a sufficiently high energy transfer between the phases. Correlograms of the results after a process time of 60 min show that higher rotor-stator speeds lead to smaller droplets. The energy density EV is the ratio of the power introduced into the emulsion and its volume flow. According to the energy density concept, the relationship x1.2 ~ EV^-b is well established. For paraffin wax the Sauter diameter x1.2 is correlated to the rotor-stator speed with a correlation coefficient of -0.73 while for montan wax the correlation coefficient is -0.12. Smaller droplet sizes can generally be achieved for paraffin wax than for montan wax, due to the lower viscosity ratio λ=ηOil/ηemulsion.

While the energy density has the expected effect on the PSD, the influence of the emulsion composition is more complex. The dispersed phase fraction is positively correlated with particle size x1.2 and span. Particles get larger and PSDs get broader for higher dispersed phase fractions. Different emulsifier quantities show that higher emulsifier concentrations (in relation to the dispersed phase) do not necessarily lead to a stabilisation of smaller particles. In fact, there are ideal concentration ranges that do not initially depend on the critical micelle concentration.

The development of the PSD over the process time is determined by the turbulent droplet break-up, which largely depends on the energy transfer from the outer phase (water) to the dispersed phase (oil). Consequently, the parameter dependencies of the development of the PSD are less clearly defined than for the product after 60 min.

The PSDs develop from bimodal to almost monomodal distributions. Typically, the distribution widths first increase before decreasing again. The achievable minimum values for distribution width and droplet size vary depending on the operating parameters. Since emulsions and melt emulsions are dynamic systems, the relationships between stress conditions, system composition and resulting product properties are complex.

The studies are funded by German Research Foundation (DFG) in the framework of priority program 2364 “Autonomous processes in particle technology – Development and implementation of concepts for model-based control of particulate processes” (project number 504809428). Financial support is gratefully acknowledged.

Poly dispersed nano-particles under the influence of varying magnetic field

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Abstract

This abstract provides an extended discrete element method (XDEM) based influence of varying magnetic field for poly dispersed nano-sized magnetic particles. Ferro magnetic particles of different magnetic properties and variations in size are dispersed freely initially and then under the influence of magnetic field in single direction to multi-directions are explored. The chain formation effect when the particles have similar properties and multi-properties are assessed. The dynamics of chain formation in vacuum and when dispersed in a base media is investigated. The computational time of each simulation case purely depends on the type of material properties and the base medium.

Content

The Complexity of Food Powders: Impact of Composition and Particle Morphology on Final Product Properties

Dr. Sheila Ruiz Barbero PhD; Lydia Karavasili M.Sc.;
Dr. Davoud Zare PhD; Dr. Vincent Meunier PhD
Nestlé Research

Abstract

Morphology is known to impact multiple powder properties such as reconstitution or flowability. However, the many different components present in food systems have a direct impact on the microstructure of the powder due to their arrangement during drying. Therefore, the complexity on determining the driving factors of powder properties such as reconstitution relays on the link between composition and microstructure.

This study aims to enhance the understanding of the relationship between composition and morphology, thereby allowing for improved control over properties such as reconstitution.

Methods: Evaluation of physical-chemistry properties within this study covers particle structure and morphology (e.g. particle size distribution, density), physical properties (e.g. reconstitution, wettability) and powder composition (chemical characterization). Model systems will be used to better understand the complexity of these systems.

Results: Smaller particle sizes and irregular shapes may have a faster dissolution kinetics due to increased interfacial area. On the other hand, the composition of food powders encompasses various components and each of them can influence the reconstitution process in different ways, e.g. carbohydrates can affect the drying kinetics due to a change in viscosity as well as the solubility of the final powder, impacting the dissolution and texture of the reconstituted product.

Conclusions: Understanding how the composition and microstructure of food powders impact properties such as reconstitution is essential for product developers and consumers and by optimizing them, it is possible to enhance the sensory attributes of the reconstituted product.

Content

Enhancing particle retention and multiphase mixing performance in an up-flow AD reactor

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[2] University of Galway, Ireland

Abstract

Provision of controlled environment is one of the fundamental factors in anaerobic digestion (AD) process. Introduction of up-flow reactors has significantly contributed to improve the AD technology, however, retention of sludge particles is a critical challenge in such reactors due to high up-flow velocity and momentum transfer caused by gas bubbles. Wash-out of sludge particles negatively impact the digestion process. Hence, it is essential to optimize reactor design to ensure proper mixing in the bioreaction zone while maintaining a sufficient sludge particle concentration. In the current work, a comprehensive multiphase Eulerian-Eulerian based CFD model has been developed by considering three phases, i.e., water (as wastewater), solids (as sludge particles) and gas (as biogas bubbles). Three different design configurations of AD reactor were evaluated through this model. The results indicated that modifying the reaction zone with a baffle arrangement can contribute in recirculation and affective mixing of multiphase flows. Sludge holding capacity reaches to approximately 80% with the top baffled reactor whereas configuration of an additional baffle at location of 500 mm from the reactor floor resulted in retention of up to 90% of the sludge particles. As an outlook of this work, further studies are planned to optimize the reactor performance with respect to different operating parameters such as inlet velocities, sludge particle density and the sludge bed height.

Content

Synthesis of particle-based silica aerogels and their pharmaceutical use as drug carrier systems

Jennifer Pierick; Dr. Viktor Maurer; Dr. rer. nat. Jan Henrik Finke; Prof. Dr. Georg Garnweitner
Technische Universität Braunschweig

Abstract

Promising active pharmaceutical ingredients (APIs) often exhibit poor aqueous solubility and thus a low bioavailability that substantially limits their pharmaceutical application. To address these challenges, such APIs can be incorporated into highly porous carrier systems that stabilize their amorphous phase, facilitate a fast dissolution and therefore improve pharmacokinetics. Silica aerogels (SA) synthesized via a precursor-based sol-gel approach have been investigated for this purpose. Here, a new particle-based synthesis strategy that leads to aerogels with widened morphological and structural variety is applied and evaluated with respect to its suitability for pharmaceutical applications.

SA were prepared via a controlled, salt-induced destabilization of aqueous silica nanosuspensions followed by a supercritical drying step. It is shown that the gelation process is mainly dependent on particle size, solid fraction and added salt concentration. The resulting SA possessed different porosities and pore structures contingent on particle size and manufacturing parameters, which in turn are proven to have a tremendous effect on the drug carrier performance for the model drug ibuprofen.

For example, investigating several drug loading strategies along the process chain, post-synthetic co-milling was identified as the one with best loading capacities enabling amorphous incorporation of 25 wt.-%. In addition, an enlarged specific surface area caused by reduction of the primary particle size led to improved loading efficiencies of respective SA. SA-based powder formulations also indicated an accelerated release behavior compared to the crystalline form (up to 40x). Furthermore, loaded SA were compacted into tablets using various compaction stresses.

Content

Flame-made calcium phosphate nanoparticles: synthesis, surface modification and delivery of antimicrobial biological molecules towards healing bacterial skin wound infections

Dr. Niki Karouta PhD; Prof. Dr. Georgios Sotiriou
Karolinska Institutet

Abstract

Antibiotic-resistant bacterial infections pose a critical global concern, particularly in skin wound cases, necessitating innovative alternatives to traditional antibiotics. The prolonged inflammatory phase, connected to untreated bacterial infections, impedes proper skin healing, lowering patients' quality of life and placing immense burdens on healthcare systems. Antimicrobial biological drugs, also known as biologics, including enzymes and peptides, offer antibiotic-free solutions combating antimicrobial resistance with minimal immunological risks. However, their delivery faces challenges such as short circulation half-life and susceptibility to environmental factors.

To address these limitations, this work explores hybrid nanoformulations (HNs) incorporating biocompatible calcium phosphate (CaP) nanoparticles synthesized using Flame Spray Pyrolysis (FSP). CaP nanoparticles, known for their excellent biocompatibility, biodegradability, and low toxicity, are designed as nanocarriers to stabilize and protect biologics while enhancing their antimicrobial efficacy. The use of FSP enables fractal-like structure, high surface area and scalable, reproducible production of CaP nanoparticles. This study focuses on hybrid formulations optimizing the synthesis parameters, refining the design of the hybrid nanoformulations for biologics stabilization and delivery, and conducting extensive physicochemical characterization. The antimicrobial activity of LL-37 and Lysozyme in the HNs is evaluated both in vitro, using MRSA bacterial cultures, and ex vivo, in fresh human skin samples. This project aims to provide critical insights into the parameters for loading biologics onto CaP nanocarriers and to expedite their translation to clinical applications as therapeutic nanoplatforms.

Content

Prefunctionalization of minerals by dry grinding in the presence of Punicines for selective separation

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Abstract

Punicines are phenolpyridinium compounds suitable for surface modification of lithium aluminate and other mineral particles due to their versatile chemical functionalization without losing switchability. They exist as cations in acidic solutions, neutral dipolar molecules around pH 7, and as anions or dianions in basic conditions. Light can reversibly convert punicines into radicals, enabling external control over their binding to particles and their surface properties via pH and light.

For efficient separation of valuable mineral components from gangue materials, particles are ground to the micrometer range. This comminution creates new surfaces and induces chemical and structural changes and increases surface reactivity. Adding punicines during comminution ensures uniform surface binding, resulting in higher separation efficiencies compared to dry or wet mixing.

Three separation mechanisms are considered: forced triboelectrification (FTC), selective wet agglomeration, and flotation.

FTC involves applying high voltage to a conductive surface interacting with the material, neutralizing target material charge while others remain charged. This enables separation in an electrostatic separator.

Selective wet agglomeration employs a suspension liquid, an oil-based binding liquid, and suspended particles. Hydrophobized particles form agglomerates via oil droplet adhesion. The influence of punicines on adhesion forces is studied using a FluidFM® system.

Flotation separates valuable particles via selective adsorption of air bubbles. Punicines act as collectors, hydrophobizing particles and enhancing their affinity for air bubbles. A feasibility study with lithium aluminate demonstrated significantly higher yields when punicines were added during co-grinding.

Content

Micro-CT-based computational workflow for multidimensional particle systems and separation process characterization

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Abstract

Tromp functions, commonly used for the quantitative description of separation behavior in particle processing, typically consider solely individual particle descriptors. However, because separation behavior is often influenced by multiple descriptors, multivariate Tromp functions are necessary to describe their joint influence on separation. We present a workflow for computing multivariate parametric Tromp functions [1]. This involves processing microscopic image data of particle systems before and after separation, statistical image analysis and multivariate stochastic modeling [2]. The workflow is applied to quantitatively characterize the magnetic separation behavior of Li-bearing minerals, including quartz, topaz, zinnwaldite, and muscovite, using micro-computed tomography (micro-CT) and scanning electron microscopy with energy-dispersive X-ray spectroscopy (SEM-EDS) analysis. Key particle descriptors such as volume-equivalent diameter, zinnwaldite volume fraction, flatness and sphericity are examined as factors influencing separation behavior. The study demonstrates that multivariate Tromp functions, supported by advanced imaging and modeling techniques, provide valuable insights into the complex dynamics of particle separation processes.

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Content

Impact of Particle Morphology on Forced Triboelectric Charging Behavior: A Case Study with Manganese Oxide Particles

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Abstract

Triboelectric charging, a process in which materials acquire charge through frictional contact, plays a crucial role in industrial applications, particularly in separation and recycling processes. In this study, we introduce a novel approach called Forced Triboelectric Charging (FTC). By applying a high voltage to a surface (chute) and introducing particles to interact with the wall (chute), the charge of the target material can be selectively shifted to a neutral state. Subsequently, an electric field (separator) is used to sort and separate neutral particles from charged ones. Understanding the charge mechanisms and the factors influencing particle charging behavior is vital for enhancing the efficiency of recovery process. In this study, we focus on the effect of synthesis temperature and phase variations of manganese oxide particles on their triboelectric charging behavior. Manganese oxide particles were synthesized at five distinct temperatures of 350, 550, 750, 950, and 1100°C. The temperature-dependent synthesis produced manganese oxide particles with varying crystalline phases, including MnO, MnO_2, Mn_2O_3, and Mn_3O_4, each exhibiting distinct porosities and surface areas. The results show that manganese oxide particles synthesized at higher temperatures, with reduced porosity and surface area, exhibited lower charge accumulation in triboelectric charging experiments. The phase variations also played a significant role, as different manganese oxide phases displayed unique charge-transfer properties due to their differing electronic structures and surface characteristics. Results show the influence of synthesis conditions and phase transitions on the triboelectric behavior of materials.

Content

Optimizing process parameters for different dismantling levels in the mechanical battery recycling process

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Abstract

In recent years, the development and promotion of electric vehicles and renewable energy technologies leads to a continuously growing number of Lithium-Ion batteries (LIBs). Accordingly, the amount of spent LIB will also increase in the near future. Used LIB are environmentally harmful and imply a safety risk. However, they present an opportunity to gain valuable resources like lithium, nickel and cobalt. Especially, the supply of the latter might be critical in future, that is why the development of independent secondary sources is mandatory, i.e. the recycling of LIBs.
Nowadays, the recycling focuses on the recovery of the most valuable materials of the battery. More recently, approaches are shifted to a holistic recycling process combining a hydro- and pyrometallurgical part with a mechanical pre-treatment, recovering and reuse all components for a new LIB.
In a mechanical recycling process route, the LIBs are crushed, dried and the received material is sieved and separated into several fractions. Due to many different components and primary particles of a battery, a sharp separation of the fractions is quite challenging. Dismantling of battery modules on a cell level reduces the total amount of components and by this also the impurities in the recovered black mass and the effort of separation of all recyclable materials. An efficient mechanical recycling route on module and cell level for recovering the maximum black mass is presented. The impact of the process parameters for different dismantling levels on characteristic properties like particle size, composition, particle morphology and yield of the black mass is illustrated and analyzed. Furthermore, variation of the rotor speed profoundly influences the recovered black mass, its characteristics and yield.

Content

Transformer Models in Particle Technolgy

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Abstract

Transformer-based Large Language Models are reshaping interactions with digital and physical systems. Originating in natural language processing, they leverage large-scale pretraining and flexible token embeddings (text, numbers, images, etc.) for reasoning and cross-domain applicability—now extending into particle technology.
This talk introduces key concepts of the architecture, illustrating how attention mechanisms maintain context across long sequences and how model scaling can yield emergent problem-solving abilities. We then explore practical applications:

- Fine-Tuning: By adjusting a small portion of parameters (e.g., via LoRA/QLoRA), experts can efficiently tailor generic LLMs to specialized tasks in particle engineering.
- Retrieval-Augmented Generation (RAG): Models query external document repositories or experimental data, producing grounded, up-to-date answers while reducing “hallucinations” and enhancing factual reliability.
- Mixture of Experts (MoE) & Agent Frameworks: Multiple sub-models or agents address specialized tasks. “Chain-of-thought” steps and self-consistency checks help mitigate errors, and advanced agents autonomously plan sub-goals, run simulations, and refine solutions. These methods were tested on domain-relevant questions, including exam-level material in particle technology.
- Autonomous CFD Agents: Integrating with CFD, specialized agents automate tasks like mesh creation, parameter setup, simulation execution, and post-processing, here at the example of a reactor in Ansys Fluent. This reduces manual workload while enabling rapid design-of-experiment cycles and on-the-fly optimization in process engineering.

This research is funded by the DFG under SPP 2364.

Content

Development of a New Particle Charge Classifying Chamber

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Abstract

The classification and measurement of particle charges are fundamental in various disciplines, including pharmaceuticals, and material sciences. The precise measurement of particle charges is critical for processes such as drug formulation, and the development of novel materials. Currently, there are several instruments available for measuring particle charge, such as the Faraday cup electrometer (measures only the net charge) and the electrostatic separator (Nabiulin & Domashnev, 1965), which is is designed for large particles.

In this work, we present a newly developed particle charge classifying chamber, designed to measure the charge of particles with sizes ranging from 50 µm to approximately 1 mm. A minute sample of particles is dispersed into a cyclone, where they acquire a charge upon contact with the cyclone wall. These charged particles then enter a chamber where a transverse electric field is applied. The particles' landing positions, influenced by their charge, size and density, as well as the electric field strength, are mapped. The system is modelled using Computational Fluid Dynamics (CFD) and Lagrangian particle tracking simulations coupled with an empirical charge transfer model. By varying the operating conditions, the simulations generate data for training a machine learning model. This model (an ensemble of the Decision Tree, Random Forest, and shallow Artificial Neural Network) learns to interpret the deposition patterns of the particles based of their charge distribution. This novel approach integrates advanced simulation techniques and machine learning to provide a more efficient solution for particle charge measurement, fostering a deeper understanding of particle behaviour and interactions across multiple scientific and industrial applications.

Content

Packing properties of spherical, nanoporous, and deformable gel particles

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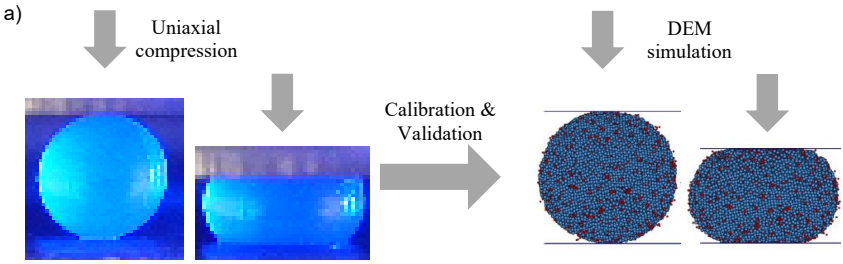
Abstract

Biopolymer aerogels are extremely light, highly porous (up to 99%) materials with remarkable properties. They possess large specific surface areas of up to 1200 m²/g and low densities of approx. 0.1 g/cm³. These characteristics, combined with the high availability and biocompatibility of the raw materials, have positioned biopolymer aerogels as an area of intensive investigations over the past decade. Their various applications include carrier matrices for flavors and active ingredients, catalyst supports, and thermal insulation materials, etc. Thus, the global demand for aerogels has experienced continuous growth, intensifying the need for optimization and scale-up of the production process.
The synthesis process typically encompasses three distinct stages: (1) gelation of an aqueous biopolymer-sol to form a hydrogel network, (2) solvent exchange with an organic solvent, usually ethanol, and (3) supercritical CO_2 drying to convert the alcogel into a highly porous aerogel. The production of aerogels in the form of millimeter-sized particles offers significant advantages in handling and production efficiency, particularly when compared to alternative geometries.

Currently, a batch-wise solvent exchange and supercritical drying of wet gel particles in packed beds is a promising solution to meet the economic requirements of the aerogel production. However, the weight of the particles and external forces (such as fluid flow) can deform them, thereby influencing the macroscopic shape, microstructure and process operation, limiting the operational process window. Therefore, this study aims to evaluate the impact of process steps, due to their variable solvents, on the mechanical properties of biopolymer-based aerogels. To achieve this, force-distance as well as strain-stress curves were recorded during uniaxial

compression tests of individual gel particles and small-scale packed beds (100 - 600 particles). First results indicate a significant increase in resistance to mechanical stresses as the material converts from hydrogels to alcogels and ultimately to aerogels.

Additionally, DEM simulations employing a tetrapod model approach were conducted (Fig. 1a) to improve the understanding and prediction of solvent release during uniaxial compression beginning with the soft and deformable hydrogel state. This approach enables a more accurate assessment of solvent effects during the early stages of the aerogel production. For this purpose, the force–strain curves of the hydrogel particles derived from simulations are calibrated and validated using experimental data (Fig. 1b), showing good agreement with the measurements. In addition, the Poisson's ratio and associated deformation behavior of the particles were systematically analyzed and evaluated. A further advantage of the simulation approach is the ability to quantify water release, which is represented as blue spheres in the simulations. While this aspect is experimentally difficult to assess, it significantly affects the mechanical properties of the particles and thus influences their final performance in the aerogel production process. Future work will aim to extend these investigations to alcogel particles, enabling the simulation of the solvent exchange step in the production process.



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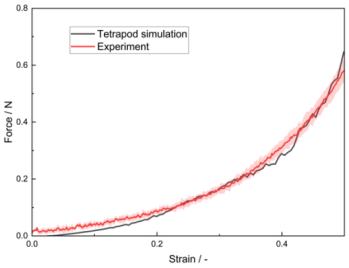

Fig. 1: a) Schematic representation to illustrate the procedure for a DEM simulation with the tetrapod approach of a hydrogel particle under uniaxial compression (the right panel shows the tetrapod's (red) representing the gel-network and sphere meso-particles (blue) that are used to represent enclosed water within the pores) b) tetrapod simulation results of the force-strain curve of hydrogel beads under uniaxial compression in comparison to the experimental data for a loading speed of 3 mm/s.

Despite the current challenges, batch-wise production of aerogels in a packed bed is considered as a promising approach to meet economic demands. However, future work is needed to improve our understanding of the gel's deformation behavior under various process conditions.

This research project is funded by the German Research Foundation (DFG, GRK 2462: "Processes in natural and technical particle-fluid systems" (PintPFS).

Probing the underlying mechanisms of the triboelectric effect with Density Functional Theory

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Abstract

The triboelectric effect is a ubiquitous phenomenon with significant implications for powder processing and particle technology. First observed by the ancient Greeks, it continues to present challenges across industries. Triboelectric charging can cause particle adhesion on heated vessel walls, leading to “sheeting,” destabilize formulations through agglomeration, and even trigger electrostatic discharges, posing risks of fires and explosions. Despite extensive research, the fundamental mechanisms driving this phenomenon remain debated. There is no consensus on the dominant charge transfer species, whether electrons, ions, or nanoscale material fragments.

Recently, interest in harnessing the triboelectric effect for practical applications, such as powering small devices, sensors, and harvesting ambient mechanical energy, has driven a surge in research. This has led to an increased use of density functional theory (DFT) calculations, opening new pathways for exploring triboelectric charging mechanisms at the nanoscale.

In this work, we employ DFT to unravel electron transfer mechanisms within particulate systems. We propose novel methods to probe the charging behaviour of individual functional groups, enabling molecular design, constructing first-principles-based triboelectric series, and examining the effects of mechanical stress and strain gradients on charge transfer. Additionally, we illustrate how DFT complements experiments, particularly in analysing the impacts of humidity and antistatic agents on triboelectric charge transfer.

By leveraging first-principles calculations, this work seeks to deepen understanding of the causes of triboelectric charging and inspire strategies to mitigate uncontrolled charge accumulation during powder and particulate processing.

Numerical investigation of wet particle collision dynamics

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Abstract

In many large-scale processes employing dense gas-fluidized beds with liquid injection, agglomeration might occur due to the adhesive forces exerted by the liquid on the particles. To unravel the complex dynamics of agglomeration, it is key to understand the interactions of wet particles. In this study the collision of fully wetted particles is studied numerically, extending the previously reported studies for partially wetted particle collisions (Shao et al, 2022; Fan et al, 2024). To validate our numerical simulations the experiments of Bunke et al,2024 on fully wetted particle collisions are used.

In this research, Direct Numerical Simulations are employed to simulate this multiphase system. The gas-liquid interface is represented using the Local Front Reconstruction Method based on the work of Shin et al,2011. The fluid-solid interaction is represented using the second-order ghost cell Immersed Boundary Method from Deen et al, 2012. The positions of the particles are updated by solving Newton's equations of motion. The drag force is calculated based on the method of Deen et al,2012 whereas the collision is treated with the hard-sphere approach from Hoomans et al,1996.

From the simulations, it has been established that increasing both the liquid layer thickness and the liquid viscosity results in lower apparent restitution coefficients. This is explained by the higher degree of viscous dissipation when the liquid bridge is extended. Additional factors being considered in our simulations are the rotation of the particles and the impact angle.

This publication is part of the project Understanding of wet agglomeration of non-spherical particles in fluidized beds with project number 19951 of the Open Technology Programme which is financed by the Dutch Research Council.

Content

Evaluation of powder flow stability using powder discharge test by pressurized fluidization

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Abstract

For example, power plants use pressurized fluidized bed powder feeders to supply fuel powder. However, when the fuel powder is discharged, unstable powder flow occurs. Powder characteristics such as flowability and cohesiveness are believed to affect unstable flow. Therefore, it is necessary to understand the powder flow stability and feeding characteristics under conditions where fluidizing air and pressure are generated simultaneously in the powder bed. In this study, we fabricated pressurized fluidized powder discharge test equipment and measured the powder discharge mass, the interstitial air pressure acting on the powder bed, and the powder flow pattern. As the experimental conditions for the pressurized fluidization discharge test, air velocities at the top and the bottom of the powder bed were changed based on the minimum fluidization velocity. The materials used were glass beads, foam glass beads, and pulverized coal; these belong to the Geldart A particle. The glass beads have high flowability, while foam glass beads and pulverized coal have low flowability and high cohesiveness. As a result, the glass beads with high flowability showed a mass flow regime in a vessel and stable discharge characteristics with a constant mass flow rate. On the other hand, it was confirmed that rathole and funnel flow occurred with foam glass beads and pulverized coal, which have low flowability. Then, the mass flow rate of powder and interstitial air pressure at this time showed unstable flows that fluctuated with time. These results suggest that the stability of powder flow with different flowability and cohesiveness can be evaluated by the fluctuation of the powder discharge mass and the pressure acting on the powder.

Content

Enhancing Flowability of Surface-Functionalised Glass Beads through Aerosil Coating

Prof. Mikio Yoshida PhD[1]; Wei Pin Goh PhD[2]; Prof. Dr. Mojtaba Ghadiri PhD[2]
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Abstract

In various industrial applications, the flowability of particulate materials is crucial for efficient processing and handling. Coating particles with glidants can significantly improve their flow characteristics, reducing issues such as clogging and inconsistent flow rate. The uniformity of coating is affected by the surface functional groups of both the particles and glidants. This study focuses on the coating of surface-functionalized glass beads (host particles in the size range 70-110 μm), obtained through acid washing (hydrophilic) and silanization (hydrophobic), with Aerosil (guest particles), a widely used glidant. Two types of Aerosil: hydrophobic and hydrophilic, both within the same size range, are used.
The flowability of the coated glass beads was assessed using the FT4 rheometer, which measures the work required to penetrate a rotating impeller into a powder bed and withdraw it, referred to as the specific downward and upward flow energy. Scanning Electron Microscopy (SEM) was conducted to assess the surface coverage of Aerosil on the glass beads. By experimenting with different compositions, we also aimed to determine the optimal concentrations for each type of Aerosil. The results reveal distinct differences in the flow behaviour of the glass beads depending on the type of Aerosil used as well as the type of glass surface that the Aerosil is coated on. These findings highlight the importance of selecting appropriate glidant types and concentrations to enhance the flow properties of particulate materials. Understanding these interactions can lead to improvements in the efficiency and reliability of industrial processes involving particulate materials.

Content

Scale-bridging, correlative lab-based X-ray micro- and nano-computed tomography for quantitative analysis of complex, hierarchical particle systems

Allison Götz[1]; Fabian Lutter[2]; Dennis Simon Possart[2]; Daniel Augsburger[2]; Usman Arslan[2]; Carmen Rubach[1]; Moritz Buwen[1]; Sabrina Pechmann[2]; Umair Sultan[3]; Alexander Kichigin[1]; Nora Vorlaufer[4]; Tor Hildebrand[5]; Peter Suter[5]; Prof. Peter Felfer[4]; Prof. Nicol

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Abstract

Porous, particulate materials of complex hierarchical structures are vital in devices like batteries, catalysts and stationary phase materials. Their properties depend on factors such as morphology & porosity, making the characterization of particle and pore statistics, like particle & pore sizes, tortuosity and porosity, essential. Pore sizes range from micro- (< 2 nm) to meso- (< 50 nm) and macropores (> 50 nm), while particle sizes vary from nanometers to hundreds of microns and agglomerates up to millimeters. This complexity necessitates correlative 3D microscopy for a comprehensive understanding of these systems. Tomography techniques inherently face a trade-off between volume and resolution. Transmission electron tomography (ET) can resolve nm-range features but is limited to volumes below (1 µm)³. X-ray nanotomography (nanoCT) resolves features down to 50 nm in a volume of (16 µm)³ but both methods typically focus on single particles. X-ray microtomography (microCT) allows characterization of particle agglomerates at resolutions in the micron range. To overcome the limitations of this trade-off, we propose a comprehensive 3D characterization workflow by correlating ET, nanoCT and microCT. Our approach aims at establishing a feedback loop between synthesis, characterization and data assessment, and enables the quantitative analysis of size, morphology and pore systems of individual particles, but also large structures across multiple scales. A tailored sample shape facilitates the 3D analysis adapted to each technique's field of view, enabling high-resolution characterization of small volumes while also allowing statistical studies of thousands of particles, enhancing the precision of quantitative 3D analysis through correlating data across imaging modalities.

Content

Refractive Index Determination of Particulate Systems using Single Particle Light Scattering

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Prof. Dr.-Ing. Wolfgang Peukert PhD
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Abstract

The complex refractive index of particles is a material property, depending on their composition and internal structure such as porosity. It is a function of wavelength and temperature. For various optical measurement systems, such as those that measure the scattered light from particles, it is essential to know the exact refractive index of the system to obtain accurate results. However, often the refractive index is unknown. Thus, the development of new measuring devices and methods for the refractive index determination of particulate systems is of high interest.
In our work, we use flow cytometry (LUMiSpoc, LUM GmbH), which is based on hydrodynamic focusing to align particles individually in a sample stream by means of a surrounding sheath flow and subsequently to measure their scattered light in the forward and sideward direction. If the refractive index of the spherical particles is known, their particle size distribution can be derived from the measured intensity distribution using Mie's theory.
We show how to determine the refractive index based on calculations using Mie's theory under given boundary conditions. For well-defined spherical particles, the real part of the refractive index can be determined from the average particle size or the exact particle size distribution. We also discuss for less defined polydisperse systems under which boundary conditions the refractive index can be calculated directly from the measurement signal, i.e. without any prior knowledge of the particle size. Furthermore, the influence of absorption on the resulting scattering signals is studied to determine how precisely the absorption coefficient, i.e. the imaginary part of the refractive index, can be determined for absorbing particles.

Content

Characterization of polydopamine shells on polystyrene particles by analytical buoyant density equilibrium experiments

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Friedrich-Alexander-Universität Erlangen-Nürnberg (FAU)

Abstract

Polystyrene-polydopamine (PS@PDA) core-shell particles are used as novel building blocks for colloidal photonic crystals. To advance the hierarchical complexity of this system, understanding the particles' properties in solution, especially their morphology and composition, is crucial. We implemented analytical buoyant density equilibrium (ABDE) experiments within an analytical ultracentrifuge (AUC), complemented by H2O/D2O density contrast AUC methods, to investigate the density and hydration of the PDA shell. ABDE-AUC experiments involve the in-situ formation of a density gradient within the AUC cell, achieved by using a gradient forming material, where the particles migrate to the position, where their density matches that of the surrounding medium. Moreover, density contrast AUC experiments in various H2O/D2O solvent mixtures enable the determination of the particle's anhydrous density. In our methodology, we first designed the gradients with the Hermans-Ende model. Subsequently, we determined the anhydrous and buoyant density of bare PS particles, validating our findings through a comprehensive approach that included scanning electron microscopy and sedimentation velocity measurements. Next, we moved to PS@PDA particles, synthesized under varying reaction parameters, namely the concentrations of core particles and of the dopamine monomer. We retrieved buoyant and anhydrous densities and extracted the optical spectra of the particles in the formed gradient. This combination of AUC methods allows for the assessment of critical quality attributes of the functional particles. For instance, the shell porosity, cross-linking density and water adsorption of the dopamine shell as well as effects on the optical properties of the particles can be retrieved.

Content

Thermomobility analysis (TMobA) of Airborne Nanoparticles

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Abstract

Nanoparticles behave differently from bulk materials during thermal analysis. Their electrical mobility varies with size, shape, and charge in thermal processes. Using a tandem DMA setup with an electrically heated tube furnace between two differential mobility analyzers (DMA), we measure electrical mobility as a function of temperature — a method termed thermomobility analysis (TMobA). This technique enables detailed studies of aerosol chemical reactions and nanoparticle component evaporation, benefiting from rapid heat and mass transfer in the free molecular regime.

One application is the nanoscale distillation of low-volatile sugars in mixed nanoparticles (glucose, sucrose, raffinose, and dextran). While these sugars decompose at ~300 °C in bulk, TMobA shows a stepwise decrease in particle size due to free molecular evaporation. Dextran decomposes at ~540 °C, seemingly at a higher temperature than in TGA, which is explained by the developed decomposition model. This stepwise evaporation could aid in cleaning airborne functional nanoparticles.

TMobA also reveals unique decomposition pathways for chemical reactions, such as silver nanoparticle formation from silver nitrate. Unlike in TGA of bulk silver nitrate, silver nitrate decomposes differently in the highly transient TMobA measurement, highlighting its utility for studying complex reactions like silver nanoparticle synthesis.

Content

On the structure of nanoparticle clusters: effects of long-range interactions

Rens Kamphorst[1]; Maximilian F. Theisen[1]; Ankur D. Bordoloi[1]; Prof. Dr.-Ing. Samir Salameh[2]; Dr. Gabrie M.H. Meesters[1]; Prof. Dr. J. Ruud van Ommen PhD[1]
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Abstract

The fractal structure of aggregates consisting of primary nanoparticles naturally arises during their synthesis. This is typically considered to be a fully stochastic process. However, we propose that long-range interactions, such as van der Waals forces, actively influence particle clustering, altering aggregate properties. Using an off-grid 3D model, we demonstrate that such interactions reduce cluster density and fractal dimension; effects that cannot be accurately captured in 2D models due to underestimated screening. Furthermore, we approximated the range within which van der Waals forces dominate the aggregation process. Our findings indicate that neglecting the effective range of van der Waals forces during synthesis leads to an overestimation of aggregate density by approximately 10%. While we focus on nanoparticle synthesis, our conclusions are broadly applicable to systems dominated by long-range forces, such as magnetic or electrostatic interactions, which may exert even stronger active pulls.

Green Fluorescent Protein as a Sensitive Model for Room-Temperature Biopharmaceutical Drying

Dr. Maksim Mezhericher PhD; Dr. Zehao Pan; Dr. Junshi Wang; Prof. Dr. Howard A. Stone PhD
Princeton University

Abstract

Rapid Room-Temperature Aerosol Dehydration (RTAD) is a novel, scalable technology for powderization and thermal stabilization of pharmaceutical products, utilizing fine droplets that evaporate rapidly at room temperature. This study employed Green Fluorescent Protein (GFP) as a model biological molecule to optimize RTAD system design and process parameters. We investigated the effects of droplet size, multiphase flow patterns in the drying chamber, and polysorbate 20 as a model surfactant on GFP fluorescence after drying and powder reconstitution. Experiments demonstrated that polysorbate 20 significantly influenced GFP fluorescence intensity, especially for smaller droplets. Computational Fluid Dynamics simulations revealed that GFP fluorescence intensity in dry powders depended on multiphase flow patterns in the drying chamber. Non-axisymmetric flows and closed circulating streamlines near the drying gas inlet negatively impacted fluorescence intensity. Through iterative optimization of chamber design, process parameters, and feedstock formulation, we achieved recovery of GFP fluorescence intensity exceeding 96% in the obtained dry powders compared to initial samples. This study establishes GFP as a sensitive model biologic and its fluorescence intensity as a powerful tool for rapidly assessing bioactivity following dehydration. The insights gained from this study have broad implications for the design and scale-up of room-temperature drying technologies, potentially transforming the production of dry powder biopharmaceuticals. By minimizing drying-induced stresses for thermally sensitive biologics, RTAD offers a promising alternative to conventional spray drying methods, opening new avenues for pharmaceutical manufacturing and stabilization.

Content

Revealing Triboelectric Charging in Legume Powders: In-situ analysis of environmental impacts on single particle charges

Greiner, Joshua & Foerst, Petra

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Abstract

Next to the worse environmental impact, the rapidly growing world population reinforces the search for a sustainable food supply. Due to their regional availability and high protein content, legumes and legume proteins are used for the production of meat alternatives by high-moisture extrusion and 3D printing. Recently, proteins from legumes were isolated by wet fractionation methods, consuming a high amount of water and energy. Electrostatic separation is based on triboelectric charging and is representative of the upcoming dry fractionation methods to obtain plat protein concentrates as a sustainable alternative to the wet extraction process. In contrast to wet fractionation methods, the dry ones are based on the powders' physical characteristics, such as particle size or charge. Electrostatic separation is a combined process of surface-specific triboelectric charging in turbulent particle-gas streams, followed by fractionating the charged particles in an electric field.

Triboelectric charging describes the charge transfer between particles during impact. This transfer is highly dependent on the particles' surface characteristics and the composition of the particles. Therefore, it is reported that legumes' protein and starch compartments gather different charges – protein bodies tend to charge positively, while starch granules charge negatively. Nevertheless, there is less knowledge of the charge single particles of legumes' protein and starch fraction can obtain in turbulent particle-gas streams. To understand and improve the fractionation process, it is crucial to enhance knowledge of the mechanism behind the triboelectric charging of insulators and food particles.

The introduction of a special particle tracking velocimetry setup (PTV) by Landauer et al. (2019) and Perez-Vaquero et al. (2020) made it possible to track the trajectory of charged particles in an electric field. By analyzing the deflection and size of the particles, it becomes feasible to calculate the charge of individual particles. This approach allows for the in-situ measurement of single

particle charges. The aim of the present study was to investigate how different collision and particle properties affect the charge acquired by a single particle.

The study revealed that the average specific charges of protein and starch particles are similar, with protein particles carrying a slightly higher absolute charge. The main factor influencing particle charges was found to be the flow of transport gas, despite the particle properties such as moisture and chemical composition. Transport gas velocity controls turbulent intensity during the charging process and, therefore, mainly determines the frequency of collisions between particles and particles with the wall of the charging pipe. The absolute value of particle charge increased with an increasing transport gas velocity and a Reynolds number between 2741 and 6854. A further increase in transport gas velocity results in a decrease of the particle charges absolut value accounting for a relaxation process.

Advances in Aerosol Nanostructuring: Toward Functions and Control of Next-Generation Particles

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Keywords: macroporous structures, mass transport, aerosol process

The development of functional materials is an important research issue for building a sustainable society. Fine particles play an important role as materials for solving this issue. In recent years, among fine particle materials, nanostructured particles composed of single nanoparticles as building blocks have been attracting attention as the next-generation fine particle materials due to the following reasons[1-2].

-Sub-micron size (100 nm or larger) makes handling easier than nano-size materials.
-Flexible composites of different nanoparticle materials are possible, and hybridization of functions is expected.
-Porous structure with regular pores is possible, and high specific surface area, low density, low dielectric constant, and mass transfer to the inside of particles are expected.
-Hollow structure is possible, and low density, low refractive index, low dielectric constant, endohedral function, and adiabatic property are expected to be improved.
-Core-shell structure is possible, which is expected to increase the weather resistance of the material (longer life), control the release of the encapsulated components, and reduce the use of raw materials.

On the other hand, in order to develop these nanostructured particles as innovative functional materials, it is extremely important to address the following issues i)-v).
i) Investigation of materials other than single-component oxides, ii) Quantitative analysis of the internal structure of particles, iii) Investigation of functionalization inside pores of porous structured particles, iv) Elucidation of correlation between structure and function of nanostructured particles, and v) Construction of process science for production of nanostructured particles for practical use.

The author has been involved in the development of nanostructured particles for about 10 years, and has conducted research on macroporous, hollow, and core-shell formation of microparticles by utilizing aerosol processes and colloidal self- assembly, and on the

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evaluation of their functions as materials (**Fig. 1**) [3-4]. In this presentation, we would like to introduce our recent work on the synthesis and control of advanced nanostructured particles and their application in energy and environmental fields.

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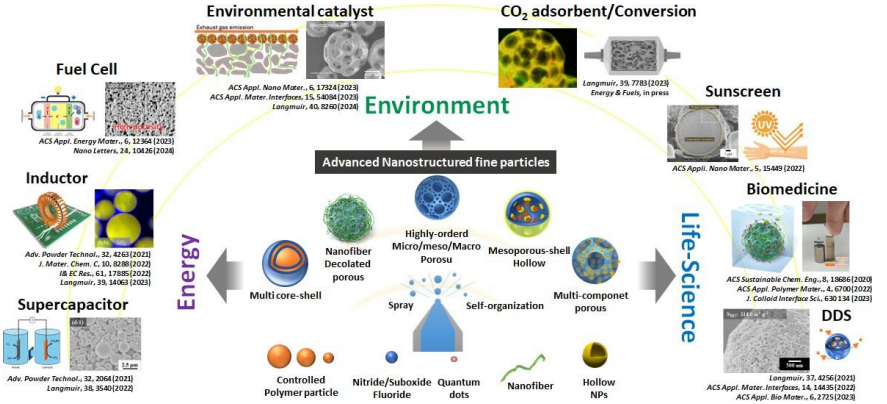

Fig. 1. Structuring of nanoparticles and their application to functional materials.

Challenges and Solutions for Data Communication in the Process Environment for Particle Size and Shape Analysis

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1. Introduction

Sympatec develops, manufactures and sells innovative particle size and shape analysis instruments for lab and process applications and offers adaptable samplers, mixers and probes with at-line, on-line, and in-line solutions [1]. These instruments rely on robust sensors and powerful software for control and communication.

Data communication is becoming increasingly important in modern process plants. Today the integration of a measuring system into a customer's communication infrastructure is highly individualised. Until now, the focus has been on transferring important measurement results to a process control system. Today, devices for analysing particle sizes independently evaluate raw data, report potential process errors in advance and, if necessary, control various supporting peripherals.

The successful integration of our instruments into various processes is achieved through the efficient incorporation of specific customer requirements for data communication into our standardised procedures.

2. PAQXOS software

Sympatec's particle size and shape analysers are supplied together with the PAQXOS control software. This software is used to perform measurements, evaluate measured raw signals and distributes the calculated results to a wide variety of receivers. PAQXOS is installed on the control computer or on a virtual PC. The PAQXOS sensor control software enables an almost arbitrarily degree of automation of all our sensors, both in the control of the measurement process and in the output of the results. All program functions can be controlled via user dialogues and are optionally also available for a high-level programming language SCRIPT working in the background.

Individual measurements with our process sensors can be performed even without programming by selecting the sampler and all parameters in the PAQXOS measurement settings. These settings can be saved as SOP (Standard Operating Procedure) for the specific sample line and product. This ensures consistent and efficient measurements.

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3. Instruments

Most of our core sensor technologies of laser diffraction, image analysis and ultrasonic extinction are also available for use in processes (see Figure 1). They have a wide range of applications, from the harshest industrial conditions to GMP environments. Accurate and reliable measurement data not only requires precise and powerful sensor technology. Another important aspect is representative sampling, to ensuring that the samples measured accurately reflect the entire material. In addition to the reproducible dispersion, which creates an even distribution of the individual particles for the measurement, these three factors form the basis for optimal analysis results.

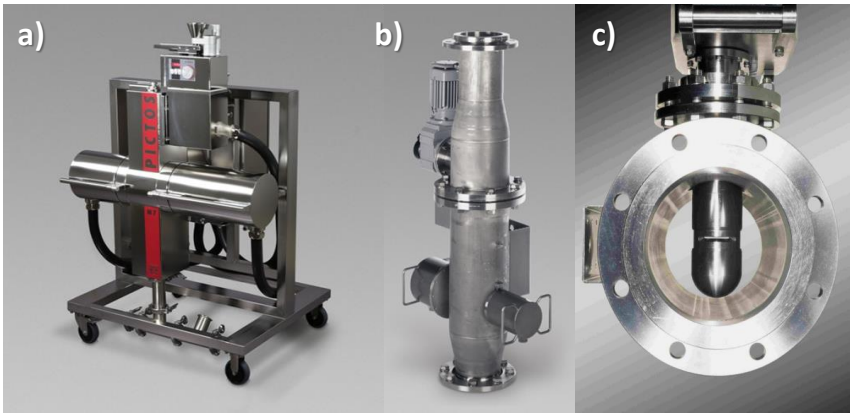

Fig. 1: Examples of process adaptation: a) PICTOS & VIBRI, image analysis, at-line; b) MYTOS & TWISTER, laser diffraction, in-line; c) OPUS, ultrasonic extinction, in-line

Another important point is the flexibility and configurability of the system. Multiple samplers can be connected to a single sensor, with the PAQXOS software fully operating the sensor and all samplers in a recently introduced standardised control concept. The parameters of the system and the measurement sequence are stored as an internal device configuration including all peripherals. These operating parameters can be configured by the user and are interpreted by PAQXOS and the system components.

4. Communication protocols

Digital field bus systems are now well established. Measurement data can not only be transmitted as floating-point numbers with high accuracy, but the correct transmission can also

be verified together with metadata using checksums and secured by encryption. There are also multi-dimensional data types and options for tracking changes. Figure 2 gives an overview of all possible communication channels that can be operated by a PAQXOS measurement computer in standard configuration without any further extensions.

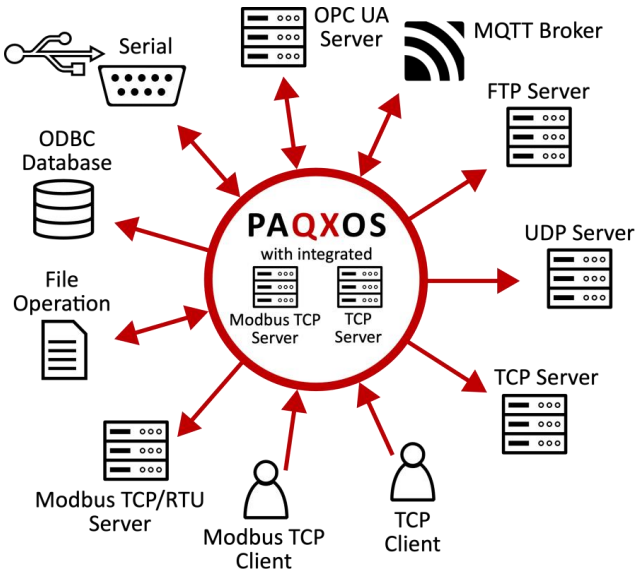

Fig. 2: Possible PAQXOS communication channels, standard configuration

PAQXOS opens up even more communication channels through technical extensions that are commonly employed in the process environment. In these cases, the software communicates with a communication module that acts as a transmission interface. Examples of such communication interfaces include programmable relays, gateways and virtual servers, which can be installed in addition to PAQXOS on the host computer or elsewhere. With PAQXOS now mastering all the main functions, a complex programmable logic controller (PLC) could be replaced by a simpler programmable I/O control module.

The SCRIPT, which controls measurement sequence of the sensor and the transmission of measurement results, can be configured in advance for the capabilities required in the field. Adaption to changed boundary conditions or customer requirements is also possible at any

time after installation. Integrated templates for standardised measurement sequences with integrated individualisation via dialogue make this procedure much more efficient.
In addition to an overview of possible technical implementations, specific installations of measurement systems for particle size analysis are analysed regarding their data communication.

5. Conclusion

Recently introduced standardised project workflows at the beginning of the installation at the customer’s site enable rapid adaptation, even if the local conditions on site change. The PAQXOS sensor control software takes over all the complex controls of the system, supplemented by the built-in programming language, which integrates the entire process communication through its standardised program library. In addition to our sensor technology, sampling and dispersion, flexible data communication has developed into the fourth key component that builds the bridge for the optimum measurement results for the customer's process application.

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Standardized and Unified Semantic Description for Particle Generation Processes

Henk Birkholz; Dr.-Ing. Norbert Riefler; Prof. Dr. Lutz Mädler PhD

Abstract

The creation of science and research data that adds value is an essential enabler for collaboration in the material science and engineering domain. Rules that guide the structuring of such data representations are a necessary forerunner. While creating specification for representations is the first step, it is also entwined with a significant threshold: how to agree on the specification and how to render them useful to communities and application in various scales.

In this talk, the process of particle generation is used as an application domain to demonstrate different approaches of creating a shared set of interoperable data representation rules. The approaches range from: stand-alone specifications, de-facto standards based on best common practices, standards based on a central registration authority for interoperatbility, and standards based on many authorities that allow for modular composition.

Approaches to structure research and science data, classic and modern data representations, and exemplary venues where corresponding standards engineering takes place are presented. Additionally, the advantages and disadvantages of central and distributed registration authorities for standards are illustrated. Before the conclusion, impact of data authenticity and believability along the post-processing value-add graph is discussed (but not as an afterthought).

Automated Particle Removal System for Lab Equipment under Vacuum: A Case Study on Draft Tube Baffle Crystallizers

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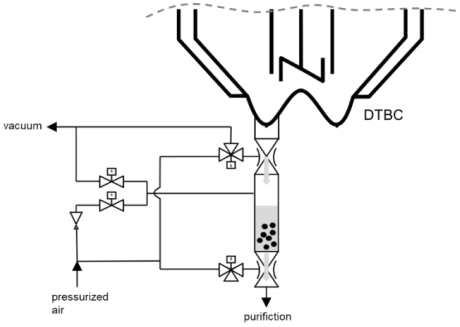
Abstract

Product removal, Vacuum Operation, Lab Scale DTBC, Continuous Operation

The continuous product removal from vacuum applications on a laboratory scale poses significant challenges, particularly for temperature-dependent crystal suspensions. These challenges derive from the small ratio of particle size to tubing diameter. In this contribution, an automated gate system is presented that enables controlled and selective product removal with minimized disruption to the crystallization process.



The gate system was specifically designed for the removal of product from a draft tube baffle crystallizer (DTBC), which has demonstrated reliability in industrial crystallization processes for producing medium to large crystals with a narrow particle size distribution [1]. To facilitate selective product removal, the classification behavior of the crystals in the DTBC was evaluated in experimental studies by determining the crystal size distributions in the product removal and in the fine grain dissolution. Thereby, the impact of different stirring speeds and crystal contents was considered for different product removal rates. Additionally, the module was integrated into the MTP standard in accordance with VDI guidelines 2658 [2], facilitating seamless integration into modular systems according to the VDI guidelines 2776 [3].

This contribution thus addresses the challenges in downscaling a DTBC for continuous operation under vacuum conditions, but is equally applicable to other continuous vacuum applications on the laboratory scale.

Content

Acknowledgement: The authors thank the German Federal Ministry for Economic Affairs and Climate Action (BMWK) for funding this research as part of the REUNION project (FKZ: 03EN2100B).

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Application of flotation and gravity separation for the recovery of lithium from slags addressing multivariate particle properties

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Abstract

Froth flotation is a widely established heterocoagulation separation process for the raw materials industry. It is most efficient for particles in the size range of 20 µm – 200 µm and is of particular importance in the context of upgrading critical materials. The growth in relevance of Li-Ion batteries, particularly for electric mobility, granted lithium the classification of a critical raw material. As a result, its recycling is essential to ensure a sustainable supply for future battery production. Froth flotation can be variably applied directly in the beneficiation of recycled battery materials or to selectively upgrade lithium from slags of pyro metallurgic recycling of batteries – currently unexploited. Slags often have a complex composition and texture challenging for selective physical separation. Small lithium aluminate crystals, the typical Li-bearing phase, lead to fine grinding for liberation, affecting flotation efficiency. So far, current flotation methods often result in insufficient lithium yields and low enrichment factors. There is a need of jointly improving the texture of slags through a concept named engineered artificial minerals (EnAMs) and the efficiency of physical separation by addressing multivariate particle properties.
Different particle property fractions exhibit varying flotation behaviors. Thus, in this study we evaluate the recoverability of individual particles in a combination of gravity separation and flotation. Particle size, shape, and composition (i.e., liberation and association) are considered. Particles are characterized with Mineral Liberation Analysis and X-Ray Powder Diffraction. This study aims to provide insights into froth flotation for improved recovery separation performance, especially for fine and complex resources.

Content

Understanding the separation of fine printed circuit board particles with X-Ray Computed Tomography and Particle-Based Separation Models

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Abstract

Particle separation is fundamental for the energy transition and raw material production. Continuous developments are needed to address the increasing complexity of geological and anthropogenic particles due to the depletion of easy-to-separate materials. For anthropogenic particles, or secondary raw materials, challenges arise from their multiphase composition, microstructure, and alloy variations, significantly affecting separation processes. Recycling printed circuit boards is particularly impacted. Finer grinding is often required, despite the associated challenges in physically separating finer particles.
This study investigates the separation of printed circuit board particles (400–630 µm) using a novel eddy-current separator, benchmarked against analytical heavy liquid separation. Thousands of particles are characterized per separation product via X-Ray Computed Tomography. A grey-value threshold differentiates voxels of glass fibers, brominated epoxy, and crushed glass from those of Cu, Ni, Au, and solder mask. Data containing five particle descriptors (equivalent sphere diameter, aspect ratio, sphericity, metal content, plastics content) is used to train particle-based separation models.
After model validation, the most relevant particle properties for each separation test are identified. Results show correlations between particle size and shape with recovery probabilities for both processes investigated. A correlation between these geometric properties cannot be ruled out. The method addresses limitations of 2D particle characterization, predicts recoverabilities, and is broadly applicable to recycling systems. Further work is recommended to refine shape descriptors for this highly variable particulate material.

Content

Recovery of tantalum through engineerable crystallization in metallurgical oxide systems

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Abstract

Low concentrations of high-tech elements often limit their efficient recovery from chemically complex municipal and industrial wastes, i.e. metallurgical slags. To tackle these challenges, the DFG Priority Program 2315 introduced the Engineered Artificial Mineral (EnAM) principle which aims at stabilizing target elements (i.e. Ta) in crystalline phases through a controlled slag composition and thermal processing, facilitating their recovery through liberation and separation. A common metallurgical waste stream worth to recycle are fayalitic slag systems (FeOx-Si2O-CaO-Al2O3), generated during copper ore refinement and the pyrometallurgical treatment of electronic and electrical waste (WEEE).
In the scope of the EnAM approach, we introduced a versatile method to identify unknown EnAM phases and potential flux elements targeting the recovery of refractory metals from a fayalitic model slag system. The method comprises a crystallographic database screening, model slag synthesis as nanoparticles using flame spray pyrolysis (FSP), laser smelting and material characterization for a systematic screening of potential flux elements that favor EnAM formations. Targeting Ta as recovery element, the database screening suggests Ca (slag constituent) and Y (new element) as promising EnAM forming elements. Indeed, the formation of Ca-Ta-O and Y-Ta-O phases during the FSP process and the associated EnAM potential were confirmed with XRD analyses. Subsequent laser melting and crystallographic analyses revealed the formation both crystalline EnAM candidates upon adding the respective element. The results will enable a correlation between thermal slag processing and phase crystallization in the form of CCT diagrams which will be essential for the application of the EnAM recycling concept.

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Sensitivity study on the effects of a composition dependent breakage criterion in discrete element method (DEM) simulations of high pressure grinding rolls (HPGR)

Paul Hirschberger; Prof. Dr.-Ing. Harald Kruggel-Emden
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Abstract

The discrete element method (DEM) has been extensively used to model and investigate comminution. For comminution modelling, the large number of particles and complex breakage mechanisms are challenging. Although computational power is rising, the computational costs of such simulations are still immense. Additionally, the materials composition and texture during breakage events has rarely been considered so far and the interactions between different material phases, which is related to this during comminution are not yet well understood. The texture of the material or particles in this context is understood as the mineral composition and spatial arrangement of phases, grains and grain boundaries to name only a few. Since experimental investigations of composition and texture related relationships in the context of comminution are complex and time-consuming, a texture inheritance model for particle breakage in the DEM has been developed [1]. The model uses spherical particles and a particle replacement method (PRM) scheme to ensure fast calculation speeds, which allows simulations with large quantities of particles, while also conserving particle texture in breakage events.
The model [1] makes use of transformations developed by Roşca et al. [2] to transform the texture of spherical particles to a cubical shape. The cubical texture can be fragmented using a size dependent breakage function without generating void spaces between fragments. To contain the cubical assembly of fragments within the spherical parent particle, fragments are uniformly shrunk to around 57% of their actual diameter. In the time steps following the breakage event the fragments grow using a growth rate limited by a maximum permitted overlap between fragments and walls or particles. Since this process can add energy to the system, it is iteratively adapted to ensure realistic particle-dynamics in the system by monitoring mass and volume losses due to shrinkage during breakage events and the number of fragments formed. The growth procedure checks if growth would result in overlaps

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exceeding the maximum permitted overlap with surrounding particles, fragments and walls using an optimized CGrid algorithm [3], similar to the contact detection.

To assign composition and its spatial distribution to particles in the simulations, particles are composed of a spherical voxel grid. Each voxel inherits a phase property that is sampled from a texture representation of the material in question. This results in one large array to store the texture information of all particles in the system in, whose size is dependent on the resolution of the particles' voxel grids. The calculations of texture inheritance in breakage events during simulations are optimized to be simple operations that adapt and pass on the position of the parent particle in that array to its fragments.

Using the described texture and texture inheritance model, the breakage behavior of a rock material is investigated in high pressure grinding rolls (HPGR). For reference of the HPGR, the simulation set-up by Cleary and Sinnott [4] is used. Initially, a homogenous material is assumed and material strength parameters by Cleary and Sinnott [4] are used. Afterwards, the material is modelled as a magnetite-iron ore following the model by Wiegel and Li [5] as a random spatial distribution of magnetite grains in a rock matrix. As a starting point, the parameters for the strength of the rock matrix are taken from Cleary and Sinnott [5] and the strength of magnetite is used for the grains. The breakage strength of a particle is then calculated based on the strengths of these two phases weighted with the volumetric part of the respective phase for each individual particle. In this way, the breakage criterion is composition dependent. Hereafter, a sensitivity study is performed, in which the material strengths are varied systematically to study the impact of material composition on comminution performance.

The material strength is modelled using a peak force breakage criterion and evaluations are made on the basis of material throughput, breakage rate, liberation degree and particle size distributions. To keep computational costs manageable, the breakage function remains independent of the particles' composition to reduce the parameter space of this sensitivity study.

The relationship between composition dependence and output particle size distribution and liberation can be used as an indication of favorable material properties for comminution and liberation of natural materials and industrial by-products such as pyrometallurgical slags. Furthermore, it is a starting point for a numerical investigation of the complex dynamics of non-random breakage.

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Evaluation and possible improvement of drag force models for stalky particles utilisable for CFD-DEM simulations

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Technische Universität Berlin

Abstract

Introduction

In the context of suspended flow processes, such as those encountered in widely utilised pneumatic conveying systems, knowledge of particle-fluid interaction forces is of significant importance. A common approach to analysing particle movement as well as particle-fluid interaction forces is to couple a particle-unresolved Computational Fluid Dynamics (CFD) method with the Discrete Element Method (DEM). The utilisation of constitutive models is obligatory for this approach, in order to ensure the reliability of numerical results. In a previous study (Markauskas et al., Powder Technol. 399 (2022) 117170), two commonly used distinct approaches for the drag force calculation (cumulated sub-sphere drag and particle drag) were employed, resulting in disparate outcomes. Moreover, it is imperative to accurately predict the particle-fluid interaction force by employing a model that is able to capture the influence of the considered particle shapes. The numerical analysis of these interaction forces for non-spherical and spherocylindrical or prolate particles has been the subject of investigation in several studies (Sanjeevi et al., Int. J. Multiph. Flow. 106 (2018) 325-337, Feng et al., Int. J. Multiph. Flow. 168 (2023) 104579, Zastawny et al., Int. J. Multiph. Flow. 39 (2012) 227-239, Chéron et al., Int. J. Multiph. Flow. 171 (2024) 104692, Laín et al., Powder Technol. 435 (2024) 119428, Laín et al., Chem. Eng. Sci. 282 (2023) 119288, Ouchene et al., Powder Technol. 303 (2016) 33-43). For these axi-symmetric particle shapes, the interaction forces only depend on a single orientation angle. Nevertheless, when considering processes involving the transportation of stalky or fibre-like flexible particles that are typically present in biomass (for example, as a substitutional renewable fuel), there are two further variables to take into account due to the particles' asymmetry: particle curvature and a secondary orientation angle. The present study employs Particle-Resolved Direct Numerical Simulations (PR-DNS) to investigate the interaction forces between individual,

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curved fibre-like particles and a fluid. Subsequently, the two major calculation approaches of drag force in DEM-CFD (cumulated sub-sphere drag and particle drag) are assessed.

Numerical Methods

The Particle-Resolved Direct Numerical Simulations are performed using the lattice Boltzmann method with a multi-relaxation time (MRT) collision operator (d'Humières et al., Phil. Trans. R. Soc. A 360 (2002), 437-451) with a D3Q19 (three dimensions, 19 directions) velocity set. Furthermore, all fluid nodes are initialised with a predefined velocity distribution that is equivalent to the inlet velocity, in addition to a uniform constant inlet velocity. In the downstream direction, the pressure anti-bounce-back (PAB) outflow (Ginzburg et al., Commun. Comput. Phys. 3 (2008) 427-478) condition is applied. In order to minimise the wall effects, a free-slip boundary condition is employed at the walls in all directions perpendicular to the driving flow direction. However, the particle-fluid boundary is modelled as a no-slip wall by the linear interpolation scheme (Bouzidi et al., Physics of fluids 13 (11) (2001) 3452–3459). This is in contrast to the quadratic interpolation scheme (Bouzidi et al., Physics of fluids 13 (11) (2001) 3452–3459) which requires more computational resources since it uses three fluid nodes instead of two. Nevertheless, it has been demonstrated that the quadratic interpolation scheme can produce analogous results to the linear interpolation when a sufficient resolution is available. The body force is implemented in the form of Mohamad et al., Int. J. Heat Mass Transf. 53 (2010) 990-996, based on the method by He et al., J. Stat. Phys. 87 (1997) 115-136.

Methods

In order to create the particles for the aforementioned simulation, the multi-sphere method was utilised to form clumps. Six distinct fibre-like, curved particles were generated, varying in curvature and aspect ratio (AR). In consideration of the aspect ratio, two values were selected for further analysis. The value AR=4 was investigated in previous studies (Sanjeevi et al., Int. J. Multiph. Flow. 106 (2018) 325-337, Feng et al., Int. J. Multiph. Flow. 168 (2023) 104579). As demonstrated by Feng et al. and Chéron et al. (Chéron et al., Int. J. Multiph. Flow. 171 (2024) 104692), the aspect ratio exerts a substantial influence on the particle-fluid interaction forces. Accordingly, an additional AR of eight was analysed. Furthermore, the curvature was defined as the reciprocal of the radius of the circumcircle that best approximates the curve at a given point. This method was employed to quantify the curvature of the respective particle. It is evident that the curvature increases in proportion to the decrease in radius. In the case of straight particles, it is obvious that curvature is non-

existent. In this study, three values for each aspect ratio were selected. The curvature of four of the six particles introduces an additional dimension of asymmetry, thereby influencing the particle-fluid interaction forces through an additional angle. In order to address this issue, the particle orientation was varied in five angles for each of the two angles (polar and azimuth). This resulted in a total of five orientations for straight particles and 25 orientations for curved particles. In order to cover a wider range of flow states, the Reynolds number was varied between 1 and 500.
Initially, in order to ensure grid independence and to minimise the impact of walls, inflow, and outflow, a series of measures were executed. A resolution study was conducted at three Reynolds numbers, and a sufficient grid resolution was identified for each of these. Furthermore, the domain sizes were varied for cases in which vortex shedding was observed. After defining the resolution, domain size and particle position, the PR-DNS was conducted for all particle types with the respective orientation and Reynolds number. The results obtained are then compared with a selection of available correlations for fibre-like particles, initially. Subsequently, the obtained data will be utilised to develop correlations based on the variables of Reynolds number, aspect ratio, orientation angles (polar, azimuth), and curvature.
Additionally, the results are compared to the drag coefficient correlation of Hölzer & Sommerfeld (A. Hölzer, M. Sommerfeld, Powder Technol. 184 (2008) 361-365), since it is frequently employed in particle-unresolved DEM-CFD and also finds applications in coupled simulations using the bonded particle model (BPM) (Markauskas et al., Powder Technol. 399 (2022) 117170).

Results

Firstly, the DNS results are compared to the drag coefficient correlation of Hölzer & Sommerfeld. This correlation shows a high degree of agreement with the DNS data for low Reynolds numbers, irrespective of particle curvature and aspect ratio. In cases where the particles' orientation is perpendicular to the flow, and in which the Reynolds number is high, the data conforms to the DNS results. This conformance is independent of both particle curvature and aspect ratio.
In recent years, a number of correlations for rod-like particles have been developed. All of these correlations are primarily based on the particle orientation. As mentioned before Feng et al. and Chéron et al. discovered a significant influence of the aspect ratio on the particle-fluid interaction forces. The results of the recent study corroborate this phenomenon. In cases with low Reynolds numbers and straight particles, these correlations are in close

agreement with the data obtained from the DNS. However, when curved particles are taken into consideration, particularly in relation to variations in the azimuth angle, notable shifts in the observed trends are evident. In the cases analysed, no correlation was found to agree well with the DNS data for curved particles over a wide range of orientations.
Although the drag coefficient's dependence on polar angle follows a similar trend for all particle types, i.e. a sine squared trend (Sanjeevi et al., Int. J. Multiph. Flow. 106 (2018) 325-337, Zastawny et al., Int. J. Multiph. Flow. 39 (2012) 227-239), the dependence of lift and torque initially follows the sine cosine trend (Sanjeevi et al., Int. J. Multiph. Flow. 106 (2018) 325-337, Zastawny et al., Int. J. Multiph. Flow. 39 (2012) 227-239) before shifting drastically to a cosine squared like trend with increasing curvature. This shift is further enhanced by altering azimuth angles. The dependency of the azimuth angle has yet to be fully elucidated and is the subject of ongoing research.

Enhancing pharmaceutical formulations and drug delivery by atomic layer deposition

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Abstract

Many pharmaceutical powders have properties that are difficult to work with during manufacturing processes and clinical applications. Quality formulations must produce stable dosage forms with clinically relevant drug loads for improved therapeutic performance. Many drugs have failed to meet these expectations and as a result, have been discontinued as ineffective at the research and development stage. Atomic layer deposition (ALD, a gas-phase coating technique) is a promising technique for tailoring pharmaceutical powders for improved formulations with high drug loading capacity and improved functionality. Drugs for oral and gastrointestinal tract delivery were coated with SiO_2, Al_2O_3 and TiO_2 in fluidized bed reactors and their functional properties were measured and compared to their uncoated counterparts. The present work demonstrates the capability of ALD coating in tailoring the wettability, flowability, stability and dispersibility of pharmaceutical powders for improved formulations and delivery into the lungs and gastrointestinal tract. Carrier-free delivery, sustained drug release, and delayed drug release are some of the noted achievements in coated drugs. Prospects of ALD in biopharmaceutical applications will seek to explore the tailoring of promising pharmaceutics for improved oxidative stability and improved wettability, dissolution and stability in their amorphous phase by ALD coating. ALD coating may offer an opportunity to achieve direct tablet compression with increased drug-loading capacity.
Keywords
Atomic layer deposition; ALD; active pharmaceutical ingredients; API; excipients; amorphous phase

Content

Experimental Investigation of Thermal Mixing of Monodisperse Particles in a Rotary Drum

Wenjing Ma PhD
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Abstract

Experimental Investigation of Thermal Mixing of Monodisperse Particles in a Rotary Drum
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Abstract
To investigate thermal transfer between particles, a novel experimental methodology was developed for determination of the thermal transfer coefficient αpp for homogeneous particles from the effective thermal conductivity λbed of the corresponding particle system. The experiment is conducted in a rotary drum and care is taken to avoid the dominant effect of wall-particle thermal transfer (i.e., by a sufficient number of particles in the drum). A plexiglass box set up outside the drum is employed to minimize thermal interaction with the environment. Hot and cold particles are initially separated and the temperature evolution of these particles from their initial positions is observed with an optical camera. This study is based on a monodisperse particle system and explores mixing based on experimental data of particles with significantly different thermal properties, namely PMMA and steel. The experimental data will be useful to adjust the pre-factor in the linear relationship between λbed and αpp. And, it will break path to the major improvement of contact heat transfer simulations of polydisperse and multivariate particle systems by thermal DEM or DEM/CFD.
Acknowledgement
This work was funded by the Deutsche Forschungsgemeinschaft (DFG, German Research Foundation) – TRR 287 – 422037413.

Content

Fluid flow velocity and temperature quantification in packed beds using phase contrast Magnetic Resonance Imaging

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Abstracts

Magnetic Resonance Imaging (MRI) enables non-invasive investigation of optically opaque packed bed reactors [1-3]. The MR signal's phase ϕ encodes quantitative information such as flow velocity and temperature which can be obtained by using radio frequency (RF) pulse sequences tailored to isolate the corresponding phase shifts ($\Delta\phi$). Here, a phase-contrast flow mapping sequence is used for the quantification of **(1)** spatially and temporally resolved fluid flow fields in fixed bed reactor-like setups built for water flow (Fig. 1 (a) top) & ethane (C_2H_6) gas flow (Fig. 1(a) bottom), and **(2)** temperature difference (ΔT) maps in a test setup (Fig. 1 (c)).

(1) Flow mapping: Two equivalent MR compatible setups (Fig. 1 (a)) were built to quantify 3D water flow and C_2H_6 gas flow velocity fields around a packed bed of cylinders. The inflow conditions were chosen to achieve similar flow conditions ($Re_p \sim 100$). The flow velocity components are obtained from the flow related $\Delta\phi$ as $v_{(x,y,z)} = (\Delta\phi_{(x,y,z)} . v_{enc})/\pi$, where v_{enc} is the maximum encoded velocity along each direction. Fig. 1(b) (left-right) show the resulting absolute velocity maps.

(2) Temperature mapping: The MR signal frequency ω of the imaged nucleus (^{1}H), decreases linearly with temperature as $\omega(T) = \alpha.\gamma.\Delta B(T)$, resulting in a temperature-induced $\Delta\phi(T)$ from which temperature difference map is obtained as $\Delta T = |\Delta\phi - \Delta\phi_{ref}|/(-\gamma.\alpha.B_0.TE)$), where α = - 0.01 ppm/°C, ΔB is the local field shift seen by the ^{1}H nucleus, Δϕ and $\Delta\phi_{ref}$ are the phase maps at two different temperatures, ($\Delta\phi_{ref}$ being the reference), γ is the gyromagnetic ratio, B_0 is the main magnetic field and TE is the signal acquisition

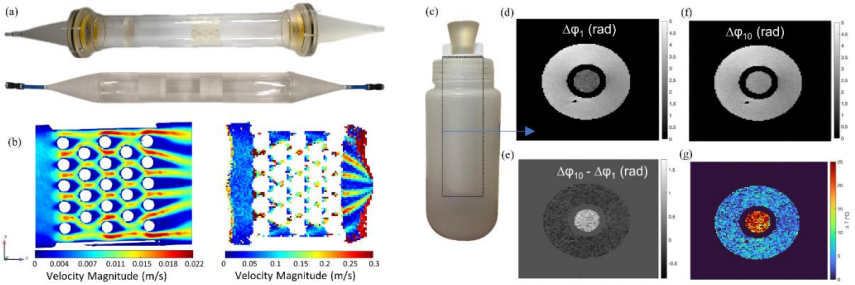

Fig. 1: (a) Flow setups for water (top) and C_2H_6 gas (bottom) flow with all components except for the plexiglass tube, 3D printed with Clear resin. (b) Absolute velocity maps in the x-z plane (z is the flow direction) of water (left) and C_2H_6 gas (right). (c) Test setup for ΔT mapping: A polypropylene bottle with a central tube 3D printed with ABS. (d),(f) Phase map $\Delta\varphi_1$ ($\Delta\varphi_{10}$) of the 1st reference (10th) measurement, respectively. (e) The phase difference $\Delta\phi_{10}$- $\Delta\phi_1$ b/w (f) and (d). (g) The temperature difference (ΔT) map.

time. Fig. 1(c) shows the setup for ΔT mapping between (i) the reference measurement ($\Delta\phi_1$) with hot water (Fig. 1 (d)) in the central white tube (~66°C) surrounded by water at ~25°C, and (ii) the same setup measured 10 times every 2 minutes. Fig. 1(f) shows $\Delta\phi_{10}$ for the 10th measurement done after 17 minutes. Fig. 1(g) shows the ΔT map obtained from the $\Delta\phi_{10} - \Delta\phi_1$ map in Fig. 1(e).

Funding : This work has been funded by the Deutsche Forschungsgemeinschaft (DFG, German Research Foundation)–422037413–CRC/TRR 287 "BULK-REACTION".

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Construction and validation of a Reactive High Temperature – Shear Testing apparatus.

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Abstract

Understanding and measuring material properties for gravity-driven particulate flow is challenging. Existing methods and devices to predict behavior are limited under extreme conditions like high temperature, pressure, and chemical reactions. Key phenomena such as friction, adhesion, and cohesion must be understood to ensure equipment functionality. Studying these phenomena under process conditions and replicating them in the laboratory is crucial to observing and measuring factors affecting particle flow.
The Reacting High Temperature-Shear Tester (RHT-ST) design, developed at the University of Salerno, replicates conventional shear testing and generates characterization parameters for particulate solids. It can operate at temperatures exceeding 1000°C and apply material compression up to approximately 2400 kPa in the gravitational direction. The shear testing unit of the equipment consists of a rotating trough that contains the sample during the test and a finned lid that creates a shear plane at a certain depth in the sample. Data from the RHT-ST, using sand, were compared with the Schulze rotating cell under various compression conditions (up to 300 kPa), showing the new instrument's applicability. Similar comparisons were made with the High Temperature-Ring Shear Tester up to 600°C, and further tests with the RHT-ST up to 800°C. The significant variation in sand's flow properties with temperature highlights the experimental procedure's effectiveness, ensuring better material understanding and process design. Consistent results confirm the RHT-ST as a robust tool, overcoming current equipment limitations and enhancing predictions of solid material flow under real-world conditions.

Content

Incipient agglomeration vs. breakage at the border to the coating

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Abstract

Agglomeration and coating are essential processes in industries such as fine chemicals, pharmaceuticals, petrochemicals, and food production. These processes involve the application of binder or coating materials onto a fluidized particle bed, coupled with simultaneous liquid evaporation. Agglomeration occurs when wet particles collide, forming liquid bridges that solidify during drying, resulting in structures resembling blueberries. In contrast, coating entails the gradual deposition of liquid solutions or materials onto particle surfaces, creating layered, onion-like structures. Although complementary, these processes often compete, profoundly influencing product structure and functional properties. Understanding and controlling the transition between agglomeration and coating is critical but challenging, as it depends on numerous factors, including equipment design, material properties, and operating conditions. Accurate prediction of this transition remains difficult, requiring extensive experimental data for refinement.
This study aims to identify the process conditions where agglomeration transitions to coating, with a specific focus on dimer formation, agglomeration kinetics, and on the other hand, the breakage of formed agglomerates. Through experimental investigations, a regime map is developed by varying key operating parameters. These findings establish critical boundaries between agglomeration and coating, enhancing our understanding of these interrelated processes and better understanding of agglomeration and breakage kinetics.

Content

Developing a process workflow to predict particle size changes in agitated filter drying

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Abstract

Active pharmaceutical ingredients are typically produced by crystallisation from solution, following which they are filtered, washed and dried. These steps are typically carried out in an agitated filter dryer (AFD), which heats the liquid through the vessel walls whilst the bed is agitated to improve heat and mass transfer. This agitation causes undesirable particle size changes due to breakage and agglomeration. The rates are dependent on process parameters (e.g., impeller speed and operation) and material properties (e.g., hardness and flowability).

In this research we aim to understand how process parameters and initial particle size distribution affect size distribution changes in AFDs, by developing and carrying out drying experiments in a small-scale jacketed vessel, which mimics the AFD process. A population balance model is coupled with differential equations for moisture evaporation and temperature rise throughout the bed, to predict particle size distribution from a given drying operation. By comparison to pilot scale results and iterating the model, we aim to develop a process workflow by which new APIs can be characterised to determine the viability of drying in an AFD and the optimum operational conditions.

Content

Mechanochemical Leaching of Valuable Ions from Engineered Artificial Minerals Using Stirred Media Milling

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Abstract

The Priority Program SPP2315, titled "Engineered Artificial Minerals (EnAM) – a geo-metallurgical tool to recycle critical elements from waste streams," is funded by the German Research Foundation and focuses on the slag phase of pyrometallurgical processes, which serves as a carrier of important, technologically critical elements. After cooling, the slag can exhibit homogeneous and amorphous structures, with crystal formation being thermodynamically driven. These crystals, referred to as "Engineered Artificial Minerals (EnAM)," act as storage media in which otherwise highly diluted elements are enriched. The goal of recycling EnAMs is to recover valuable components. Hydrometallurgical processes, such as leaching, have been established for this purpose, whereby valuable components are extracted through selective dissolution in a solvent.
This study investigates the influence of various acids and mechanochemical effects occurring during grinding on the leaching of valuable components from EnAMs. For this purpose, pre-crushed slag in the micrometer range is comminuted in a stirred media mill using water and/or different acids (HCl, H_2SO_4, HNO_3, citric acid) as leaching agents. Grinding enlarges the reactive surface and reduces the chemical inertness of crystalline phases through amorphization. To evaluate the recovery of those valuable ions, the filtrates of the suspensions sampled throughout the process are analyzed using ICP-OES. Simultaneously, particle size distribution is examined through laser diffraction measurements to determine the correlation between particle size and lithium yield and leached particles are analyzed using SEM-imaging for morphological changes. Furthermore, it's shown how process-supporting methods, such as the use of additives affect the yield.

Content

Dry particle coating of cathode active material with conductive additive using mechano-fusion process

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Abstract

The increasing demand for high-performance materials highlights the importance of dry particle coating processes in creating functionalized materials with tailored properties. The Mechano-fusion (MF) process enables the generation of hetero-aggregates by coating larger host particles with smaller guest particles, facilitating enhanced functionality [1]. This study investigates the coating of conductive additives, like carbon black, on cathode materials like NMCs (Lithium Nickel Manganese Cobalt Oxides), widely used in Lithium-ion batteries. MF experiments are conducted using the Picoline machine from Hosokawa Alpine (Augsburg, Germany) with the Picobond module, which features a stator-rotor assembly. The influence of process parameters like rotational speed and process time is studied to understand their impact on the resulting coating structures.The quality of the hetero-contact is a critical factor affecting a desired functionalized particle property, e.g. conductivity. Hence, to assess coating performance, a comprehensive methodology for 2D and 3D structural characterization is employed. Atomic force microscopy and computed tomography techniques are explored to understand the surface characteristics of coated structures. A comparative analysis with pristine materials provides insights into the influence of MF process parameters on coating quality and particle functionality. This study demonstrates the potential of MF process in producing advanced cathode materials for Lithium-ion batteries and underscores the value of multi-scale characterization techniques in understanding and optimizing coating structures.
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Streamlining Continuous Manufacturing: Wet Granulation of High-Dose Drug Formulations Without Subsequent Drying

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Abstract

Wet granulation is a vital process in pharmaceuticals, traditionally requiring drying methods that can lead to high energy consumption, prolonged processing times, and significant operational costs. These conventional drying techniques often occupy considerable floor space, creating inefficiencies in high-volume manufacturing. To overcome these challenges, we present a novel wet granulation method utilizing Potassium sodium tartrate tetrahydrate (PST) as both a water donor and acceptor, effectively eliminating the need for a separate drying step.

We evaluated this innovative approach through case studies involving Metformin Hydrochloride (MET) and Paracetamol (APAP), both high-dose active pharmaceutical ingredients. The formulations were prepared by mixing MET and APAP with Fumed Silica, followed by the addition of PST and Poly(vinyl pyrrolidone) K 30. These mixtures were processed in a twin-screw extruder at controlled temperature and feed rates. The resulting granules were characterized for particle size and tableting behavior using established methods.

Our findings indicate significant particle growth attributed to the unique properties of Kollitab® PST, which releases water of crystallization at elevated temperatures, facilitating the granulation process. The granules exhibited superior flowability and compaction behavior compared to the pure APIs, meeting compendial disintegration requirements. This method not only produces high-strength tablets with satisfactory dissolution profiles but also enhances sustainability by reducing energy consumption and simplifying continuous manufacturing processes. Overall, the use of PST in wet granulation represents a significant advancement in pharmaceutical formulation technology.

Content

Development and Testing of Bilayer Mini-Tablets

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[1] Heinrich Heine Universität

Abstract

Mini-tablets, tablets with a diameter of 4 mm or smaller, are emerging as an innovative solution for pediatric drug delivery. They provide advantages over liquid formulations, including easier transportation, lower production costs, and improved acceptability and swallowability for children aged 6 months to 6 years. Bilayer-tablets are tablets in which the dosage form consists of two layers of material with individual composition.
The aim of this study was to develop and evaluate 2 mm bilayer mini-tablets produced on a compaction simulator and on a rotary tablet press.
Bilayer mini-tablets containing Enalapril Maleate and Hydrochlorothiazide were prepared through manual granulation, followed by compaction on the Styl'One Evolution simulator using EU-B 19-tip 2 mm punch tooling .
Ibuprofen containing bilayer mini-tablets were produced at two different drug loads. The compaction process was conducted on a Korsch XM 12 rotary tablet press, utilizing 2 mm EU-D 19-tip tooling.
Critical quality attributes of the mini tablets, including mass variation, content uniformity, tensile strength, and visual layer separation were evaluated.
Achieving content uniformity in bilayer mini-tablets posed challenges, requiring adjustments in layer thickness to ensure uniform distribution. Mixing methods had varying effects on API content uniformity, emphasizing the importance of selecting the appropriate process to guarantee consistent API distribution. Visual inspection and manual mechanical testing of bilayer mini-tablets, produced using the Styl'One Evolution simulator and Korsch XM 12 rotary tablet press, confirmed distinct layer separation and strong interlayer adhesion, ensuring robust tablet performance.

Content

Influence of Particle morphology on Paint Properties – A particle Technology Perspective.

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Golcha Associated

Abstract

The morphology of industrial minerals such as talc, calcium carbonate, and calcined clay plays a pivotal role in determining their performance in paint formulations. Variations in particle size distribution and shape arise when these minerals are sourced from different locations and processed using distinct milling techniques. This study explores the influence of particle morphology on various paint properties.

Minerals with diverse morphologies exhibit different rheological behaviors, affecting parameters such as viscosity, thixotropy, suspension stability, and processing time during paint preparation. Furthermore, inherent properties such as lamellar structure and hydrophobicity contribute to enhanced barrier properties, corrosion resistance, and improved mechanical and optical performance in paints. Understanding the relationship between mineral morphology and paint performance is therefore critical for optimizing formulations to achieve desired properties at desired PVC level.

In this study, minerals were sourced from various locations and processed using milling techniques, including hammer mills, jet mills, roller mills, and ball mills, to produce a range of particle sizes and shapes. These processed minerals were evaluated in paint formulations for properties such as opacity, contrast ratio, reducing strength, acid resistance, and rheological characteristics. The findings underscore the significant impact of particle morphology on the functional attributes of decorative paints, offering valuable insights for professionals in particle technology and highlighting the interplay between mineral processing techniques and end-use performance.

Content

Investigations on the breakage behavior of legumes

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Abstract

Plant-based meat substitutes are becoming increasingly important in view of the change in eating habits in many societies. Meat substitutes are typically obtained by extrusion of melted plant protein. Legumes such as soybeans, fava beans or peas are often used as protein sources. The protein fraction has to be enriched, i.e. separated from the starch and fiber fractions, which is frequently accomplished by wet-chemical extraction of the protein by dissolving it in an alkaline medium followed by precipitation of the protein fraction in acid and drying. An alternative approach to the complex wet-chemical process is impact comminution of legumes followed by separation of the fragments into a protein-rich and a starch-rich fraction. This is possible by classification, as the protein fraction is present in domains smaller than around 5 µm, while the starch fraction in legumes forms larger domains of several 10 µm. One challenge lies in identifying suitable stress conditions to obtain high-quality (low-starch) protein flours and (low-protein) starch fractions. Although process concepts are already available for the production of protein concentrates by comminution (cf. classifier mills with deflector wheel classifier), the basic breakage behavior of legumes has not been investigated that extensively by now.
In this contribution, we present results of single particle comminution experiments of green peas (Pivum sativum) in a pin mill. We analyze the comminution kinetics as well as different shape factor distributions of the obtained fragments as a function of fundamental process parameters. Single particle comminution experiments allow for the identification of suitable stressing conditions for the disintegration of legumes.

Content

Variable volume flows in a hydrocyclone by changing the inlet geometry during the process

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Abstract

Motivation:
Hydrocyclones are designed for a specific operating point but react badly to a change in operating conditions. Reducing the flow rate results in lower centrifugal forces and hence separation efficiency. Increasing the flow rate increases the pressure loss, while the separation efficiency suffers from increased turbulence. It is also not possible to react to modifications of the material or liquid phase to be separated. The aim of the present development is to create a cyclone that can react to variable operating parameters to keep the separation efficiency at a high level.
Material & Methodology:
Good separation in cyclones requires a stable formation of the secondary vortex, which can be characterized by high tangential flow velocity (Schubert 2003). By implementing a variable cross-section in the inlet channel, the feeding velocity into the cyclone can be kept constant at changing flow rates to maintain the secondary vortex and keep comparable separation efficiency and pressure loss. An inline turbidity measurement at the upstream outlet can be used to monitor dynamically the separation efficiency. The variable cross-section area can be controlled depending on the fluctuating volume flow or on a specific outlet concentration based on the measured turbidity. Experiments showed that operating the system at a constant pressure loss improves the separation efficiency despite a reduction of the volume flow. Efficient separation at different or even fluctuating volume flow is hence possible.
References:
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Content

DEM Simulation of Impact Charge on Particles by Particle Impacts Across Different Chute Lengths

Mehran Javadi MA[1]; Dr.-Ing. Annett Wollmann PhD[2]; Prof. Dr. Alfred. P Weber[2]
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Abstract

Triboelectric charging, a phenomenon where materials acquire charge through frictional contact, is pivotal in numerous industrial applications, particularly in material separation and recycling. This study introduces a novel method known as Forced Triboelectric Charging (FTC). In FTC, a high voltage is applied to a surface (chute), and particles are introduced to interact with the wall, enabling the charge of the target material to be selectively neutralized while other materials acquire charge. Following tribocharging process, an electric field (separator) is employed to recover and separate the neutral particles from the charged ones. Understanding of the charge mechanisms and the factors that influence particle charging behavior is crucial for optimizing recovery processes. Experimental results from the FTC process revealed that chute length plays a significant role in the charging behavior. Specifically, increasing the chute length improves the specific charge on the particles, but charge saturation occurs from a certain length. To further understand this phenomenon, in this study, Discrete Element Method (DEM) simulations are employed to model triboelectric charging of particles on the chute. Simulations are conducted for chute lengths of 200, 600, 900, 1200, and 1800 mm, replicating experimental conditions. The model focuses on charge transfer during single and repeated particle impacts, to find the correlation between chute length, number of impacts, and cumulative particle charge. These simulation results are then compared with experimental data to validate and refine the understanding of the process.

Content

Influence of froth properties and particle size on the flotation separation of Li-aluminate from pyrometallurgical slags

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Abstract

Froth flotation has become increasingly important in recycling. With the growing use of batteries, lithium has been classified as a critical raw material. As a result, its recycling is essential to ensure a sustainable future supply. In addition to the direct separation of recycled battery materials, froth flotation can be applied to selectively upgrade lithium as engineered artificial minerals (EnAMs) present in slags from pyrometallurgical recycling of batteries. It has been observed that cooling and additive regime conditions facilitate the enrichment of lithium in the prominent EnAM phase, lithium aluminate embedded within the gangue mineral gehlenite. These conditions also influence the morphology, size distribution of crystallites, and the formation of additional lithium-bearing EnAMs.
In froth flotation the separation of minerals is mainly regarded to be due to selective hydrophobization of particles. However, in addition to hydrophobicity, the stability, height, and depth of the froth are critical factors influencing particle recovery and overall separation efficiency. This study explores the impact of these froth properties on selectivity and recovery regarding particle size, shape and degree of liberation, particularly focusing on the behavior of two reagent regime systems, including the surfactants punicine and oleic acid. Both regimes are found to exhibit distinct froth characteristics influencing selectivity patterns in flotation. The research examines the role of froth properties and particle entrainment, as well as the influence of fine particle liberation on the selectivity of flotation. The study aims to provide insights into the optimization of froth flotation for improved recovery and separation performance, especially for fine and complex resources.

Content

Understanding the influence of physical and chemical parameters in aqueous carrier magnetic separation of fine powder systems

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Abstract

Fine powders contained in e-waste have the potential to become an ideal source of secondary raw materials due to their high concentrations of technology metals, limited chemical complexity, and well-liberated state that eliminates the need for energy-intensive comminution. However, conventional separation methods designed for primary raw materials are typically unsuitable for such fine materials (< 10 µm). As a result, end-of-life fine powders are often landfilled, incurring financial and environmental costs while leading to the loss of critical raw materials from the resource cycle.

This study explores the separation of fluorescent lamp powders using carrier magnetic separation. The attachment affinity of magnetic carriers to individual fluorescent powders is analyzed, and the corresponding magnetic removal efficiencies are evaluated at various scales, ranging from desktop magnets to a high-gradient magnetic separator (HGF-10, Steinert). The grade and recovery of processed products are assessed through fluorescence spectroscopy and elemental analysis.

The experimental methods enable a detailed investigation of the influence of physical and chemical parameters in the aqueous medium on the separation process. Additionally, a novel method combining ion-mediated shifts in surface energies with ultrasonication is proposed to achieve complete detachment and recovery of magnetic carriers following target-powder separation.

Content

Numerical study of the dispersion behavior of CNT agglomerates in epoxy

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Abstract

It is known that the utilisation of ultrasonic technologies is an effective method to achieve the efficient dispersion of carbon nanotube agglomerates (CNTA) in an epoxy resin array. However, the concentration of nanostresses caused by the remaining agglomerates leads to a significant reduction in mechanical strength. In this work the efficient parameters for CNTA dispersion in epoxy resins during ultrasonic treatment is determined. To achieve this goal, a methodology was developed to identify the optimal conditions for high-quality dispersion, utilizing amplitude-frequency analysis of CNTA behavior in an epoxy medium, assuming that the interactions between nanotubes adhere to the Van der Waals law. For this purpose, the APROKS-CNT virtual laboratory for the automatic generation of finite element models of various types of CNTA of arbitrary shape was developed. A series of numerical experiments were performed in the ABAQUS environment to determine the critical pressure caused by an ultrasonic source, at which the CNTA in epoxy resin become unstable. The characteristics of CNTA stability loss under ultrasonic compression or shear were determined. Amplitude-frequency characteristics for various CNTA were constructed. The process of dispersion of "ARMCHAIR" type CNTs with chiral-index (6,10) in epoxy resin PHD 24 was studied, resulting in the optimal frequency and amplitude settings for the ultrasonic dispersion.
This research study is funded by the Deutsche Forschungsgemeinschaft (DFG, German Research Foundation) – Project-ID: 554884501

Content

Analyzing crown bottom break-off by means of event-based imaging

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Abstract

Droplets interacting with thin liquid layers of the same or a different fluid is a process frequently observed both in engineering applications and in nature. Splashing, as one outcome of droplet impact associated with the formation of secondary droplets, has attracted great scientific interest due to its complex and intriguing features. A particularly interesting splashing phenomenon is the detachment and disintegration of the crown originating at its base, i.e. the interface between droplet and pre-existing liquid film. Recently, the occurrence of crown bottom break-off (CBB) was attributed to the thinning of the lower crown wall with the simultaneous formation of turbulent eddies due to the strong shear field present at the crown base. These eddies generate disturbance entrained in the crown wall leading to the formation of hole nuclei, which rapidly grow and merge. In this work we employ two-perspective event-based imaging (EBI) to gain additional insights into the CBB phenomenon. In contrast to frame-based imaging, EBI detects brightness changes (events) on the pixel level, which results in an asynchronous data stream comprising the time, location and sign of the events. This gives EBI the unique quality to detect changes in a process like CBB. With our experimental setup we observe the droplet impact from a side and bottom view in order to determine time and location of the break-off process. The impact parameters such as droplet velocity, diameter and viscosity of the liquid are varied over a wide range.

Content

Comparison of pre-alloying, blending and in-situ mixing for laser metal powder deposition processing

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University of Bremen

Abstract

In the laser metal deposition (LMD) process, a high-energy laser creates a melt pool on the surface of a substrate. Metal powder is delivered through a nozzle into the melt pool and the molten material solidifies upon cooling. The process continues as the laser and nozzle move in a controlled manner, depositing material layer by layer. One critical aspect in the process is the method of powder mixing.
This study investigates the approaches to mixing powders, including in-situ mixing within the melt pool, blending powders prior to processing, and using pre-alloyed powders with the final composition. Each method presents unique advantages and challenges regarding homogeneity, microstructure control, and process efficiency. Understanding these mixing strategies is key to optimizing material performance and tailoring properties to specific applications.
Investigations were carried out for high strength low-alloyed and case-hardening steels. Pre-alloyed powders were generated by free fall gas atomization process. For prior blending as well as for in-situ mixing, commercially available powders of pure elements respectively binary or ternary alloying systems were utilized. For each material and mixing method, cubes were produced under different LMD process parameters. They were investigated regarding microstructure with focus on dissolution and distribution of the alloying elements. Hardness measurements were performed to indicate how the powder affects the mechanical properties.
The results show the influence of the powder mixing method on the properties. Dependencies between the microstructure respectively the hardness and the mixing method could be proven.

Content

Defect Formation at SS316L-Cu Powder Interfaces in PBF-LB/M Additive Manufacturing

Arvind Chouhan; Dr.-Ing. Nils Ellendt; Prof. Dr. Lutz Mädler PhD

Abstract

Multi-material laser powder bed fusion (PBF-LB/M) offers a groundbreaking approach to additive manufacturing, enabling the creation of components with tailored properties for various applications. However, processing dissimilar materials like SS316L and Cu presents challenges due to significant differences in their thermophysical properties. This study explores defect formation mechanisms in SS316L-Cu systems using numerical simulations and experimental analyses. In Cu-rich powder beds, high reflectivity and thermal conductivity lead to insufficient melt pool formation and lack-of-fusion defects at low energy densities, which are mitigated by increasing energy density. Conversely, SS316L-rich powder beds exhibit fewer fusion-related defects at low energy densities but develop keyhole porosity at higher energy densities due to keyhole instability. Steep temperature gradients across the SS316L-Cu interface cause differential thermal expansion, leading to thermal cracking, particularly at high energy inputs. A material grading strategy is proposed to minimize thermal stresses, providing insights for optimizing multi-material PBF-LB/M processes.

Stress-Based Scaling Strategy for Cathode Paste Dispersion Processes in Small-Scale Approaches

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Abstract

Lithium-ion batteries are of critical importance in a variety of electrical devices and vehicles in the present day, with their use increasing rapidly, particularly in the context of electric vehicles. In order to further increase the range of electric cars, research is being intensified on batteries, with a particular focus on the formulation and processing of the electrodes. Given the scarcity of new active materials, it is imperative to optimize smaller batches with regard to suspension and electrode properties prior to their transfer to larger batches, thereby minimizing material wastage and improving quality and robustness of transfer to large scale. [1,2]

The slurry mixing process, particularly with regard to specific energy input and the intensity of stresses acting on the particles, exerts a significant influence on the effective dispersion of materials like carbon black (CB). The decomposition of CB is critical for electrode quality, as it shapes the electrode's internal structure, influencing lithium-ion diffusion and overall cell performance. Therefore, a scaling investigation based on specific energy inputs was conducted on a laboratory dissolver to examine the impact of various processing parameters on suspension properties, across different batch sizes and energy inputs. To this end, suspensions are produced at varying batch sizes. Furthermore, the study incorporates the utilization of dissolver discs of varying diameters and heights. The specific energy inputs previously mentioned are calculated and introduced as scaling parameters for the process description. Finally, a characteristic property parameter of the suspension, such as the 'Dispersion Index of Carbon Black' (DICB) [3], is correlated with the specific energies applied at the various process parameters and batch sizes. This correlation is then subjected to physical testing and verification using a model approach to be able to estimate the specific energies and stress intensities that have been introduced in the suspension based on the choice of process parameters [4].

The subsequent process parameters are then derived with the aid of this scaling in order to predict a defined product quality for a specific batch size. The investigations are primarily

conducted on the cathode side. The demonstration is made that the DI can be deployed as a characteristic variable, and that a scalable relationship between specific energy resp. stress intensity input and CB decomposition prevails. Furthermore, it is evidenced that the selected model is capable of physically reproducing this relationship. Finally, individual suspensions are incorporated into EL-Cells to facilitate the evaluation of their electrochemical properties.

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Innovative Solutions for the Sustainable Sealing of Solids

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Abstract

Shaft seals, like the SDF-Series, are critical components in industrial processes, preventing material ingress and egress. Sustainable sealing is increasingly important to minimize material losses and enhance resource efficiency. Additionally, maintenance-friendly, energy-efficient, and wear-resistant seals help address industry challenges like labor shortages, making them essential for the economic and ecological operation of modern facilities.
Stasskol has developed innovative solutions to replace conventional stuffing box packings, with the SDF-Series as the core product for sealing solids and liquids. The design and materials of these seals can be customized for specific applications, including ATEX requirements, and are available in the CEMA standard for the North American market. The SDF-Series uses a silicone sealing ring that connects positively to the shaft, interacting with two outer sealing rings that axially seal against the housing. The use of barrier gas or a patented gasless spring connection ensures continuous, maintenance-free operation.
Extensive testing on Stasskol's test bench examined performance under varying pressures, speeds, and material compositions, including PTFE, PEEK, PE, and carbon. Results showed a correlation between frictional heat, thermal expansion, and reduced barrier gas leakage. The PTFE-based material SK133, with FDA approval, demonstrated the highest wear resistance. For high-temperature applications, carbon materials and high-temperature silicone were developed and successfully tested.
The findings confirm that the SDF-Series shaft seals not only offer innovative sealing solutions but also contribute significantly to reducing resource consumption and emissions, enabling more sustainable and efficient industrial operations.

Content

Tuning the surface chemistry of noble metal nanoparticles

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Abstract

Nobel metal nanoparticles (NPs), typically ranging in size, from 1 to 100 nanometers, exhibit unique properties distinct from their bulk counterparts. These properties make them ideal candidates for various applications, including catalysis, sensing and biomedical applications. Controlling their surface chemistry is crucial for optimizing functional performance in aforementioned applications. Our work presents strategies for the targeted modification of NP surfaces for complex nanoparticle design.
NPs are synthesized using a modified Turkevich method. Using thiol chemistry, we subsequently modify the particles' surface chemistry, allowing fine tuning of interparticle interactions and their behaviour in external environments. This facilitates the controlled assembly of complex nanoparticle ensembles by controlling particle attraction.
After modifying the surface chemistry, various characterization techniques are employed to assess and quantify refunctionalization. Transmission electron microscopy (TEM) images are used to determine the particles' core size and coupled with analytical ultracentrifugation (AUC) measurements to precisely determine the ligand shell thickness. Further, isothermal titration calorimetry (ITC) experiments and nuclear magnetic resonance (NMR) spectroscopy measurements are combined to
determine the ligand shell density on the particle surface.
Current research focuses on predictive surface design of noble metal nanoparticles using mathematical models.

Content

Surface chemistry assessment of functionalized silica materials

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Abstract

Silica materials with high surface areas such as nanoparticles and porous silicas are indispensable in many separation processes. Their surface chemistry can be adjusted by surface modification. However, the in-depth assessment of the degree of surface functionalization, molecular ordering and the resulting surface hydrophobicity/hydrophilicity, requires a combination of advanced spectroscopic and adsorption-based-techniques.
We have prepared a series of porous silica materials with different densities of trimethylsilyl groups. With solid-state 1H NMR spectroscopy under Magic Angle Spinning (MAS), absolute quantification of the functional group density was obtained. The comparison of the adsorption properties of water and argon at 87 K were used to define a hydrophilicity index. The contact angle inside the porous material was determined using novel physisorption and water intrusion methodologies.[1] We are also developing a methodology to assess the interactions of silica materials with water and other solvents by in situ solid-state NMR spectroscopy, implementing a vapor flow through the sample under MAS. Furthermore, we are developing a novel high precision in situ vapor adsorption calorimetry methodology to assess key characteristics of the pore surface including heterogeneity, energetics and adsorbate/adsorbent interactions potentials. The methodology presented here is applicable to surface chemical characterization of a wide range of silica nanoparticles and porous silica sorbents.
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Wettability characterisation of ultrafine particles in heterocoagulation separation – a special focus of the influence of particle shape

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Abstract

Froth flotation is an efficient separation process for fine particles (10 µm - 200 µm), based primarily on differences of particle wettabilities. Therefore, a fundamental understanding of the interfacial properties is required. Other particle properties, like shape and size, influence the process outcome as well. To study the impact of these particle properties on the separation, first, said particles have to be characterised thoroughly. However, there are several challenges when it comes to this, especially for the case of ultrafine particulates, where analytical methods reach their limits. Sometimes, information is not accessible at all or only by applying mathematical assumptions based on theoretical constructs.
In order to investigate the particle wettability, ultrafine (-10 µm) glass spheres and fragments are used. Their wettability is modified via an esterification reaction using alcohols, where the resulting wettability is controlled by the alcohol's alkyl chain length. Analysis by inverse gas chromatography yields information on their surface energy, which is further used to calculate the energy of interaction between said particles and cyclohexane, as this is used as a solvent in liquid-liquid extraction tests to study the particle behaviour at the interface. Furthermore, glass slides that underwent the same treatment as the glass particles, were analysed by measuring static and dynamic contact angles against water using the sessile drop method. Analysis via atomic force microscopy yields information on the hydrophobic interactions on the glass slide surface.
The correlation of the various methods shed light on their advantages and limitations in characterising particle wettability and how it is influenced by particle shape.

Content

Adapting semi-dry graphite anode processing for $LiFePO_4$ cathodes: Insights into pore structure and particle size control

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Abstract

Conventional lithium-ion battery electrode production involves a wet dispersing process to create a slurry with a low solids content, which is then coated onto a current-collector foil via slot die coating, followed by a drying step. This process results in long drying times and high-energy consumption. The granulate-based semi-dry electrode process offers a promising alternative by reducing solvent content, thereby shortening drying time and lowering energy costs, while also providing long-term storage stability. Furthermore, the reduced solvent usage allows for controlled evaporation and supports the formation of a desirable pore structure [1].

The investigation of semi-dry processed anodes showed that their distinct pore structure and distribution reduce kinetic limitations at high C-rates compared to conventionally processed anodes using planetary mixers. This improvement is attributed to higher stress intensity and stress number during extrusion, resulting in increased graphite surface area and a narrower pore size distribution [2].

This study investigates the semi-dry fabrication of lithium iron phosphate ($LiFePO_4$) cathodes, examining how different LFP particle sizes affect pore structure and lithium-ion diffusion. The higher specific surface area and distinct morphology of LFP compared to graphite elevates energy input during extrusion. A uniform coating was observed at higher temperatures, thus increasing solvent evaporation and the resulting pore formation. Electrochemical impedance spectroscopy assessed ionic resistance and tortuosity, while cyclic voltammetry provided insight into ion diffusion kinetics. SEM supported the structural and electrochemical results.

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Content

Effect of advanced infrared drying on the particulate structure of battery cathodes

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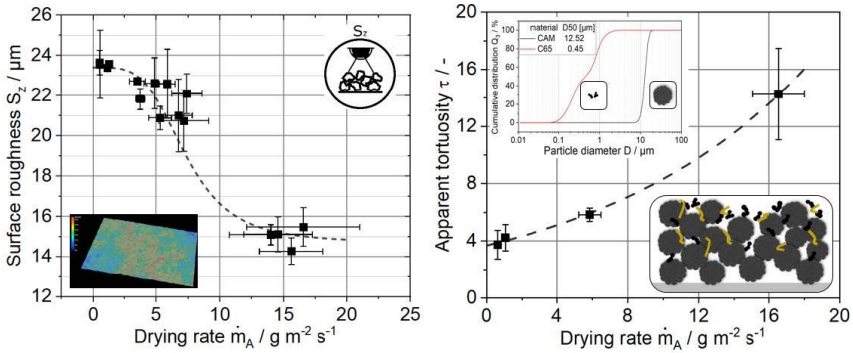
Abstract

Currently, state-of-the-art battery electrode production uses a solvent-based film coating process that requires an energy-intensive drying step. Recent studies have explored innovative drying technologies that promise to increase yield and reduce energy consumption, thereby lowering the costs and carbon emissions associated with battery production [1]. However, the impact of these drying methods on the particle structure of battery electrodes, which is critical to electrode quality and performance, has not yet been investigated in detail. While segregation of components by rapid drying have been discussed in the literature, they have not been thoroughly evaluated in the context of these advanced technologies.

This study investigates the effects of infrared drying on NMP-based cathodes. Key performance metrics are evaluated, including adhesion strength (a measure of mechanical integrity) and ionic resistance (reflecting apparent tortuosity). In addition, we measure surface roughness and identified a blocking of surface pores by inactive electrode components (binder and conductive additive), which may explain the increase in apparent tortuosity associated with higher drying rates.



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Prediction of primary particle numbers in aggregate from 2D data based on machine learning method

Rui Wang

Abstract

PREDICTION OF PRIMARY PARTICLE NUMBERS IN AGGREGATE FROM 2D DATA BASED ON MACHINE LEARNING METHOD
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The number of primary particles in an aggregate plays a crucial role in determining its morphology and characteristics. Variations in particle numbers can significantly alter the size, structure, and properties of aggregates, impacting their dispersibility, optical characteristics, conductivity, and catalytic behavior.

In this work, an artificial neural network (ANN) approach is applied to predict the number of primary particles in an aggregate from 2D data. We first generated a range of 3D aggregates with varying fractal dimensions and numbers of primary particles through a numerical model. These 3D aggregates were then rotated and projected to obtain the maximum projected area while accounting for the overlap between primary particles. Next, the 2D fractal parameters of these images were extracted using the box-counting method, and the size distribution of the primary particles was estimated based on the Circular Hough Transform method. A total of 390 sets of 2D data were employed for training the ANN model: 319 sets for training, 58 for testing, and 59 for validation. The ANN model achieved an R^2 value of 0.93 between the predicted and actual primary particle numbers, demonstrating its effectiveness in estimating particle numbers from 2D image data. Lastly, we compared the ANN-predicted results with those obtained from previous and commonly used method under three different fractal dimensions, showing that the ANN provided more accurate predictions.

Content

Development of a PET-like system for particle tracking

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Abstract

Introduction and Setup
Experimental insight into moving granular material in industrial processes as rotary kilns or grate systems, has proven to be difficult. A promising, well-established method to determine particle trajectories in various systems is positron emission particle tracking (PEPT). The detector systems usually used for PEPT are expensive medical positron emission tomography (PET) scanners, built from granular inorganic scintil-lator crystal arrays. A cost-effective alternative setup is an array, consisting of plastic scintillator bars read out at both ends. In the planned experimental setup, four walls, each consisting of 22 scintillator bars, surround a generic grate system with dimensions of 300 x 300 x 320 mm3 to be investigated.
Method
Tracer particles filled with positron emitting isotopes will be traced along their paths through the granular assembly. The annihilation of a positron, emitted by the traced particle, with an electron produces a pair of two 511 keV photons, which are emitted back-to-back and detected by the scintillator walls. The posi-tions of the tracer particles will be determined by the time-of-flight difference of the two photons of each generated pair.
Detailed Geant 4 simulations have been done to find the most promising detector setup. An initial feasi-bility study, derived from performance tests on prototype scintillator bars, resulted in a spatial resolution of about 5 mm with an efficiency of 1.4 %.
Outlook
The scintillator bars, photo detectors, and read out electronic is built and tested.
Further improvements will be a refinement of the tracking algorithms and a finishing of the experimental setup by implementing constant fraction discriminators.
Acknowledgement
TRR 287, Funded by Deutsche Forschungsgemeinschaft (DFG)

Content

Laser Diffraction for Particle Size Analysis in Inhaler Applications

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1. Introduction

Laser Diffraction (LD) has long been a key method in particle size analysis, providing reliable measurements across a wide particle size range, from millimetre-sized granules to submicron particles. Standardised by ISO 13320 [1], LD uses Fraunhofer and Mie theories to derive particle size distributions from light diffraction and scattering. Recent advancements, such as wide-angle detection systems with polarisation-separated light detection (developed in the BMBF-funded project AutoKlass, FKZ 033RK065C), have enhanced LD's ability to resolve fine particles, enabling precise measurements in both wet and dry applications.

These improvements have broadened LD's application scope, especially in sectors requiring precise particle size characterisation. In the pharmaceutical industry, LD plays a crucial role in developing and ensuring the quality of inhaler devices like nebulizers, Dry Powder Inhalers (DPIs), Metered Dose Inhalers (MDIs), and superfine mist inhalers (e.g. Soft Mist™ Inhaler, SMIs). Accurate particle size analysis guarantees that these devices deliver the correct dosage and particle size, optimising drug delivery to the lungs for particles ranging from 0.3 µm to 6 µm, the ideal size range for lung deposition.

In this paper we introduce new developments for particle size analysis for inhaler applications based on laser diffraction systems including modifications of the INHALER setup, especially related to the measuring chamber, and enhanced software tools for segmented particle size analysis (especially relevant for spray and inhaler applications).

2. Laser Diffraction with Sympatec's HELOS + INHALER

The combination of our LD instrument HELOS together with the INHALER dispersing system (see Fig. 1 (left) for our compact HELOS/BR instrument with five different measuring ranges covering a particle size range from 0.1 – 875µm) offers rapid, non-invasive measurement capabilities, allowing for precise analysis of small and costly sample volumes, including fine aerosols. This enables continuous quality control during production, ensuring particle size distributions remain within specified ranges to meet regulatory standards. LD also supports long-duration measurements of fine particles from nebulizers over several minutes, making it

Content

indispensable for inhaler development and fine particle applications in pharmaceutical manufacturing.
A specific advantage of the laser diffraction technique is that the size measurement itself is independent of the air flow rate. This enables testing under real breathing simulation, which is crucial for the development and optimisation of inhalers, as the inhalation manoeuvre can influence particle size and distribution.

Although the European Pharmacopoeia [2] focusses on the measurement of aerodynamic particle size via impaction, it also states that laser diffraction can be used as an analytical method, provided it is validated against a multistage liquid impactor, an Andersen cascade impactor or a next generation impactor. In contrast to measurement at the impactor, it is not necessary to remove the carrier particles with laser diffraction, as the entire particle size range can be detected. For a validation against existing impactors, however, it is necessary to specify the effect of the pre-separator stages.

Fig. 1: (left) Laser Diffraction system HELOS/BR + dispersing system INHALER; (right) INHALER with Dry Powder Inhaler (DPI) device (orange) adapted in front of the 90° glass pipe bend (as simulation of the throat).

3. Inhaler Applications

Inhalers are widely used devices for administering medication to the lungs. Depending on the available formulation of the drug, different driving forces are used for administration. Typical Inhaler applications are listed in Fig. 2 with Metered Dose Inhaler (MDIs), Dry Powder Inhaler (DPIs), nebulizers and superfine mist inhaler (e.g. Soft Mist™ Inhaler, SMIs). To a certain extent e-cigarettes (E-Cig.) have to be included in this listing because of the “vaping” instead of the “smoking” effect of the E-Cig. liquid vapor produced and inhaled to the human lung.

Our standard INHALER dispersing system (see Fig. 1 (right) together with a DPI device adapted) has been used for particle size analysis since many years, especially focusing on DPI inhalers with coarser particle size distributions ranging significantly above 10µm up to 200µm. DPIs contain the active ingredient in dry form and are activated by the user's inhalation. An interactive powder mixture is usually used in these inhalers. Significantly larger carrier particles (40-200 µm) are added to the active ingredient, which is usually < 10 µm in size, to improve the flow and dosage of the micronized active ingredient [3]. The agglomerated mixture of active ingredient and carrier particles is deagglomerated by the energy introduced during inhalation. As a result, the small drug particles can reach deep areas of the lungs, while the carrier particles are deposited in the mouth and throat. A high inhalation speed leads to better deagglomeration but also to earlier deposition of the particles in the lung.

Fig. 2: Inhaler applications: (left) Metered Dose Inhaler (MDI), (second left) Dry Powder Inhaler (DPI), (second right) Nebulizer, (right) Soft Mist™ Inhaler (SMI).

4. New Measuring Chamber

Applications of MDIs and nebulizers with smaller air flow rate and especially superfine mist inhalers (SMIs) are gaining more and more importance in the pharmaceutical industry the last years, strengthening the need for reliable PSA with on the one hand relatively small volume flow rates down to 1 L/min (the upper limit is up to 100 L/min) and on the other hand the need of measuring particle size distributions (PSDs) below 10µm and even below 1µm down to the nanometre regime in order to distinguish the small differences in the fine particle contributions. There are many of parameters which can significantly influence the result of the PSA, e.g. the main air flow rate of the aerosol, the temperature, humidity and pressure difference in the measuring chamber and also the conditions of the flow stream by regulating secondary air conditioning and extraction forces. In order to better include these devices in PSA for LD, a new measurement chamber has been designed and developed, embedded in the corresponding funded federal project [4] to allow for all the new circumstances including

suitable secondary air conditioning, low air flow rates, temperature and humidity control, extraction control and others.

The objective of the new measuring chamber has been to prevent particle deposition on optical components and ensure precise positioning of particles within the laser beam. The system has been designed to accommodate air flow rates ranging from 1 to 100 L/min. A coupled CFD-DEM (Computational Fluid Dynamics - Discrete Element Method) simulation methodology has been applied to effectively optimize fluid flow and particle movement.

The development of the new measuring chamber together with an enhanced control of main and secondary air flow, also applicable for air flow rates from 100 L/min down to 1 L/min allows for the controlled investigations of all Inhaler applications together with the software option of segmented measurements, described in the following section.

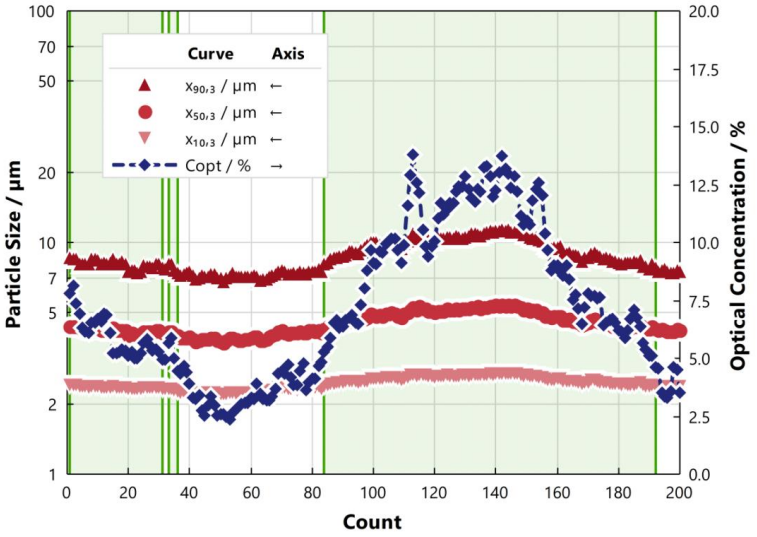

Fig. 3: Time dependent view of the optical concentration (dark blue) in comparison to the characteristic particle sizes (x_{10}, x_{50}, x_{90}). Each measurement point is a segment within the segmented particle size measurement of nebulizer water droplets. The green-shaded areas indicate the ranges of valid segments lying within the pre-defined tolerance limits, e.g. for the optical concentration within 5% - 20% for the individual segments of the measurements. Only segments within this green-shaded area are used for the evaluation of the total measurement.

5. Segmented Particle Size Analysis

Sympatec's sensor control software PAQXOS for Laser Diffraction and the optional evaluation mode SPRAY for segmented particle size analysis offer the ability to visualise the time-dependent and rapidly changing aerosol flow conditions of spray and inhaler applications.

The evaluation of the results can be changed/modified even after measurement by selecting valid and deselecting non-valid individual segments of the total measurement. This can be done by introducing tolerance limits for certain parameters, e.g. optical concentration, to check if the measured individual segments are lying within these limits or not. All valid segments taken for evaluation can be marked by green area shadings in the plots (see Fig. 3) to make the valid zones clearly visible.

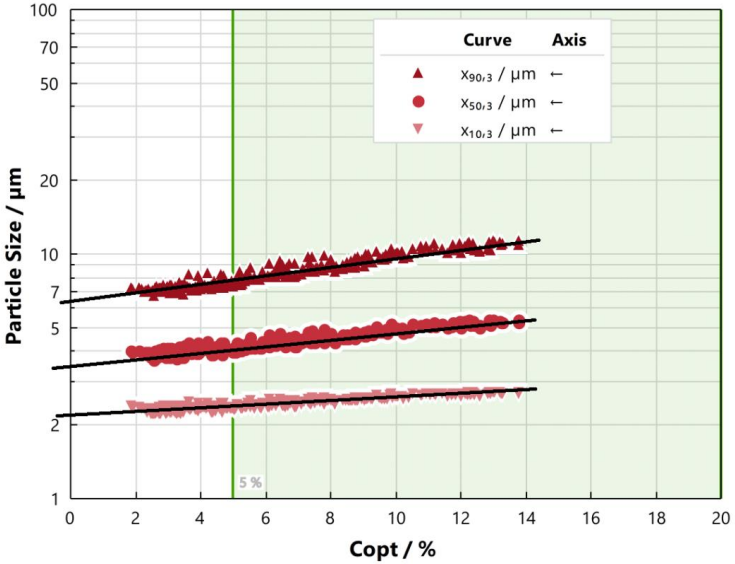

Fig. 4: Results for nebulizer: PSA analysis (TREND diagram) with variation of the optical concentration (achieved by changing the air flow rate manually) with the time-dependent behaviour shown in Fig. 3.

Time-dependent changes of the characteristic particle size values in the valid zones can then be investigated if these are time-dependent effects or depending on the parameter chosen for the tolerance limits (e.g. optical concentration). As an example (see Fig. 3) the variation of the

optical concentration (Copt) by changing the air flow rate manually leads to changing values of the characteristic particle sizes for measuring a commercial nebulizer. The green-shaded area shows the valid segments fulfilling pre-defined tolerance limits for the optical concentration within 5% and 20%. In Fig. 4 the data obtained from Fig. 3 are rearranged that the characteristic values (e.g. x_{10}, x_{50} and x_{90}) of all measured segments are plotted within the TREND diagram against the optical concentration, which has been variably changed (corresponding to the air flow rate of the aerosol through the measuring chamber). An almost linear dependency of the characteristic particle sizes (x_{10}, x_{50}, x_{90}) on the optical concentration is obtained, indicating a primary PSD for completely diluted nebulized air flows at Copt=0%. The green-shaded area shows the valid segments range as described above, indicating that the tolerance limits of Copt can be extended towards smaller Copt values, e.g. down to 2.5% for valid segments included in the total measurement evaluation.

6. Results for Nebulizer

By using commercially available nebulizer the new measuring chamber has been tested on functionality. The nebulizer (Medisana® IN 525, filled with pure water) was placed in a custom-made silicone mouthpiece for measurement. With controlled main and secondary air conditioning variation of the optical constant by changing the air flow rate show only slight dependence of the characteristic values on the optical concentration both in the time-dependent TREND diagram (see Fig. 5, top) as well as in the TREND diagram with the optical concentration as parameter on the x-axis (see Fig. 5, bottom). In comparison to Fig. 4, where the dependency on the optical concentration is clearly visible, the characteristic values only change slightly in the optical concentration range within 3% - 20% with this device. The huge changes in the optical concentration are only slightly affecting the characteristic particle size values x_{10}, x_{50} and x_{90}, indicating that the air flow rate does not influence the particle size of the water droplets significantly.

In Fig. 6 the comparison of the PSA results for different nebulizer devices measured with the new measuring chamber is shown. A commercial nebulizer (Medisana® IN 525) was filled with pure water in comparison to a mixture of water containing 4% salt (“Emser salt”) to distinguish different loads for the nebulizer. The measurements were carried out at 1.7 L/min for the nebulizer device with a similar secondary air conditioning.

Additionally, results of a commercial e-cigarette are shown for comparison. For the E-Cig. the mechanism of sucking was established by rapidly changing the volume flow rate by closing secondary air valve in front of the extraction device (vacuum).

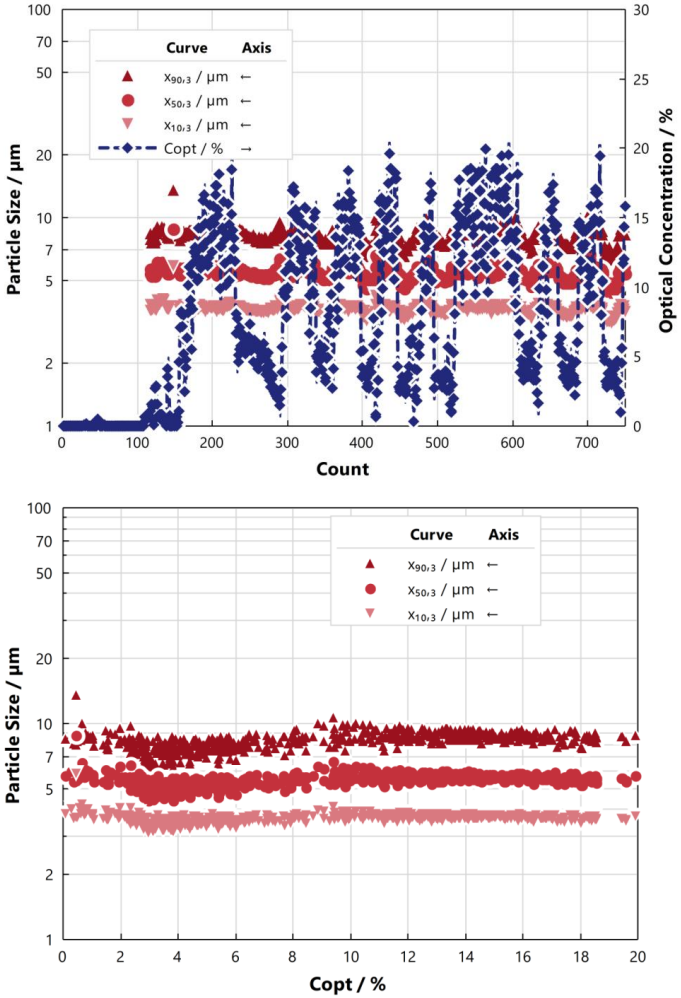

Fig. 5: (top) Variation of the optical concentration by variation of the air flow rate during measurement of nebulizer water droplets (Medisana®). The slight changes of the characteristic particle sizes (x_{10}, x_{50}, x_{90}) in dependence of the optical concentration is plotted using the segmented PSA option (bottom). In this case the particle size is almost independent of the optical concentration used and therefore almost independent of the air flow rate applied.

All results are indicating that the PSDs of the different inhalers cover the aimed size range for lung deposition ranging from below 1 µm up to 10 µm.

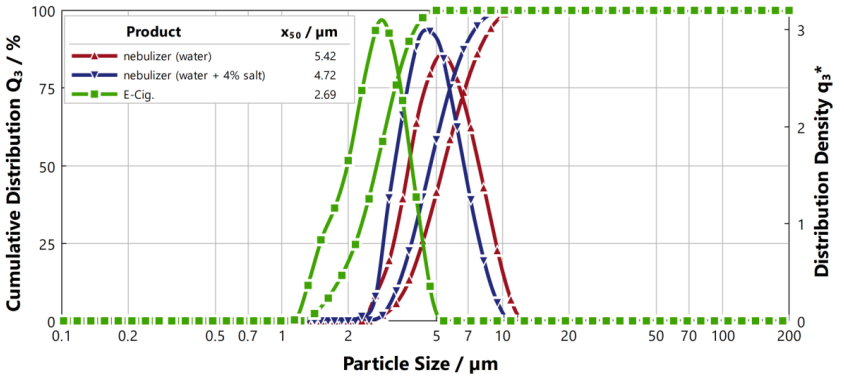

Fig. 6: Results for PSA with LD for different nebulizers: commercial nebulizer (Medisana®) with pure water compared with mixture of water containing 4% salt together with a commercial E-cigarette (VooPoo Vinci), all of them indicating the suitable size range for lung deposition ranging from 1 µm to 10 µm.

7. Conclusions

Laser Diffraction is a rapid, non-invasive method for PSA of inhaler applications, allowing for precise analysis of nebulizers, DPIs, MDIs and superfine mist inhalers. The development of a new measuring chamber together with the software option of segmented particle size measurements offers the possibility to extend the range of air flow rates down to 1 L/min for investigating pharmaceutical inhaler applications with demanding air flow conditioning and breathing simulations for adequate simulation of the lung deposition of the applied drug.

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Effective density of shear sensitive aerosol particles in the con-text of dustiness measurement

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Abstract

The dustiness of bulk materials is a key physico-chemical property for assessing potential risks to human health or environment. It is described by the weight fractions of an aerosolised powder that are i) inhaled through nose and mouth, ii) penetrate beyond the larynx and iii) penetrate into the alveolar region. Standardised measurement methods of dustiness fractions rely on the deposition of aerosol particles in classifying filter foams after weak powder agita-tion. These methods have proven inappropriate for the dustiness characterisation of shear sensitive powders such as carbon black or fumed silica, because the fluiddynamic stresses acting on the aerosol dust particles are i) relatively high and ii) differ between the various size fractions.

A recently proposed new approach of dustiness measurement avoids these pitfalls by per-forming particle size analysis with optical measurement techniques at dust particles being generated by gentle mechanical treatment and exposed to a low-shear air flow. The optical measurement ensures that all dust fractions experience the same stress. However, it also yields geometric particle sizes, whereas the dustiness fractions are specified by their inertial deposition. Hence, the applicability of new method relies on the knowledge of the particles' effective density.

This work explores experimental approaches for determining the effective density of aerosol particles in the range from 1 µm to 100 µm. They compare geometric particle properties with settling velocity, relaxation time or inertial deposition – either for single particles or for specified fractions of the particle system. The approaches are discussed with respect to reproducibility and working ranges. Finally, the consistency of derived density values is assessed.

Content

Evaluating the effect of temperature on powder flowability

Dr. Salvatore Pillitteri PhD; Dr. Aurélien Neveu PhD; Filip Francqui
Granutools

Abstract

Numerous applications involve processing granular materials or powders at elevated temperatures. This can be due to requirements of the process or variations of the environmental conditions especially for production sites located around the world. Temperature elevation can induce different mechanisms that will change the powder properties such as moisture evaporation, change in particle properties (stiffness, shape roughness,…), or can induce oxidation… These modifications in the powder change the powder's cohesiveness which impacts its flowability. Therefore, powder flowability must be evaluated as much as possible at a temperature close to the one in the process to provide reliable predictions.

In this study, we investigated the influence of temperature on four different powders used in various applications such as powder bed fusion processes for Additive Manufacturing (AM), or dry coating processes in battery production. The flowability and the rheology of the powders are evaluated in a rotating drum geometry (GranuDrum HT, Granutools). The Dynamic Cohesive Index metric computed by the instrument is used to evaluate the influence of temperature on the cohesive behavior of the materials. Different effects of temperature on powder properties are highlighted with these typical materials. These effects are due to different phenomena that are presented to better understand physical mechanisms depending on temperature. The combination of an adequate metric with a precise temperature control opens the way for better spreadability quantification, close to the process conditions, and a better understanding of the effect of temperature on powder behaviour.

Content

Small-Angle X-ray Scattering: A Powerful Tool for structural characterization of particulate and porous material systems

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Abstract

Reliable measurement of structural features in particulate and highly porous materials is essential for materials development. Understanding specific surface area, particle size, pore size, and pore volume provides valuable insights, enabling correlations and predictions of key physical properties, such as thermal, electrical, and mechanical behavior.
When it comes to particle, pore size and specific surface area analysis, there are a plethora of available methods, such as gas adsorption, mercury porosimetry, dynamic light scattering or conventional electron microscopy.
However, small angle X-ray scattering offers significant advantages over these traditional methods, since it enables simultaneous determination of particle size, pore size, and specific surface area in a non-destructive manner. It requires minimal sample preparation, provides rapid measurements, and allows for in situ experiments to observe real-time changes.
Furthermore, SAXS covers a broad size range, bridging gaps left by techniques like dynamic light scattering or microscopy, and avoids using hazardous materials such as mercury.
These attributes make SAXS a versatile and efficient tool for characterizing particulate 2-phase systems, from liquid suspensions to metal alloys, as well as porous materials.
With the increasing number of laboratory instruments and an expanding user community, SAXS is becoming more accessible, even to non-experts. The recent publication of standards for particle size determination (ISO 17867) and specific surface area measurement (ISO 20804), along with ongoing efforts to develop additional SAXS-related standards, has extended its relevance beyond fundamental science.

Investigation Into Effect of Surface Texture on Flowability Of Battery Materials

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Abstract

Surface texture in powder flow cannot be overstated, particularly in industries such as battery manufacturing, where precise formulation is crucial. The surface texture of powder particles significantly influences their interaction and movement, ultimately impacting the overall flowability of the formulation. This research paper addresses the critical role of characterising the flowability of precursors in the context of formulation processes. The primary objective is to discern the influence of various surface textures on the flow properties of these materials. The research employs detailed analytical techniques, including the analysis of particle physical properties, cohesion assessment, and predictive flowability studies, to explore the correlation between surface texture and the flow behaviour of battery powders.
The study aims to contribute valuable insights that can be applied to optimise battery manufacturing processes. By enhancing our understanding of the intricate relationship between surface texture and powder flow behaviour, the research endeavours to provide critical information crucial for the efficient formulation and handling of battery materials. This investigation holds significance for advancing the scientific comprehension of how the surface characteristics of battery powders impact their handling, processing, and, consequently, the overall manufacturing of formulations.
Keywords: Surface texture; Particle interaction; Analytical techniques; Particle physical properties; battery formulation processes

Content

Development of powder characterization protocol for vibration-assisted dry powder printing

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Abstract

Novel Dry Powder Printed is a Binder-Jetting based technique, developed by researchers in Cranfield University. The purpose of this method was to develop a technique allowing Additive Manufacturing (AM) of Energetic Materials in a safe manner. Cranfield's Vibration-assisted Dry Powder Printer offers a series of advantages, that make it not only suitable for the defence sector but could also be used in pharmaceutical/biomedical sciences, where powdered material is used.

Unlike other powder-based AM methods, it does not distribute the powder throughout the whole powder bed – instead, powder is dispensed on demand, to produce the desired geometry. This feature makes it ideal for applications in which too much of loose, dry powder might create a hazard (i.e. energetic materials, explosive atmosphere), but also when the material available is limited and cannot be cross-contaminated. Additionally, it creates opportunities for multi-material, gradient structures, that would be particularly useful for the pharmaceutical sector, i.e. patient-tailored medicine.

During development of such a system, researchers have reported some key issues with powder flow and, as a result of it, low density (high porosity) and inhomogeneous internal structures of printed objects.

Therefore, a thorough investigation of powder rheological properties is required to further develop the method. Understanding how powders behave whilst processing them (printing) is crucial. For that, various powders have been studied to discover, how properties such as (but not limited to) particle size, shape and powder moisture content translate to the ease of printing.

Defining which physical and mechanical properties of powders are relevant in terms of vibration-assisted powder dispensing will establish the proper powder characterization protocol for this technique. A well-defined protocol for material testing will ensure repeatability and efficiency of the method.

Content

Jet assisted fluidized bed production of battery hetero aggregates with structural analysis

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Abstract

All-Solid-State-Batteries (ASSBs) are set to provide high energy densities along with a safer energy storage medium than conventional batteries, which could become crucial in the electrification of transport systems. One of the main challenges of ASSBs is the upscaling of the production process. To that end, the use of a vibrating fluidized bed is proposed for the mixing, coating and aggregation of the battery materials. Thus, the fluidization and mixing behaviour cathode material is investigated in a vibrated fluidized bed with pulsated gas flow and a high velocity microjet. The microjet enhances the fluidization and also plays a major role in the creation of hetero agglomerates or aggregates that consist of multiple species.
A parametric study including vibration parameters and pre-conditioning of the particles via drying or sieving was explored, while the fluidization characteristics were investigated. Lastly, microjet parameters were varied. Electrochemical properties such as maximum capacity, electronic powder conductivity and cycling stability were investigated.
It was shown that the vibration-, jet- parameters influence the fluidization characteristics. Further, the mixing quality improved when using a microjet during fluidization. The successful production of composite material was confirmed. Lastly, the improvement of electrochemical properties and the creation of heteroaggregates was confirmed.
Funding of this work by the German Research Foundation (DFG) in the framework of Priority Programme 2289 "Hetero Aggregates" is gratefully acknowledged.

Microwave Plasma-Synthesized Graphene-Based Platinum Single Atom Catalyst for Electrochemical Application

Prof. Dr. Hartmut Wiggers; Aydan Akyildiz-Mert
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Abstract

The development of efficient and sustainable catalysts is essential for addressing global challenges in energy, environmental sustainability, and chemical production. Single Atom Catalysts (SACs) with their isolated active metal centers offer exceptional atomic efficiency, catalytic performance, and selectivity. Among SACs, platinum-based catalysts stand out for their stability under harsh conditions and outstanding activity in reactions such as oxygen reduction (ORR) and hydrogen evolution (HER). When combined with few-layer graphene (FLG) – a material known for its high electrical conductivity, large surface area, and chemical stability – Pt-SACs become highly promising candidates for energy conversion and storage applications, benefiting from synergistic effects like enhanced catalytic activity and improved charge transfer, which can be further enhanced by doping the FLG with sulfur.
In this study, we present a one-step synthesis method for sulfur and platinum-doped few-layer graphene using a microwave plasma reactor. Starting from vaporized precursors, this innovative technique allows rapid, energy-efficient processing with precise control over doping levels and atomic dispersion. The simultaneous incorporation of sulfur and platinum under controlled plasma conditions ensures a uniform dopant distribution and strong synergistic interactions. Structural and chemical analyses, including TEM, XPS, and Raman spectroscopy confirm successful doping and the preservation of the graphene framework and catalytic tests of the materials produced will be shown. This scalable, environmentally friendly synthesis approach provides a promising pathway for the development of next-generation catalysts, advancing renewable energy technologies.

Content

Modeling and Optimization of Heterojunction Contributions in Metal Oxide Gas Sensors

Luca Völkl; Dr Xiao-Xue Wang; Dr. Florian Krause; Prof. Dr. Andreas Rosenauer PhD; Prof. Dr. Lutz Mädler PhD; Prof. Udo Weimar; Dr. Nicolae Bârsan

Abstract

This project focuses on modeling the impact of heterojunctions on gas sensing performance. Sensors composed of various semiconducting metal oxides (SMOX) are fabricated using flame spray pyrolysis (FSP), employing configurations such as hetero-aggregates formed via double-nozzle FSP, bilayered materials with heterojunctions at their interfaces, and overlapping layers where each material connects to its respective electrode. Numerical simulations are first developed for individual n-type and p-type grains and are then extended to include the anisotype heterojunction formed at their interface. Material properties required for the model are derived from literature work function data and experimental measurements of work function changes. To realistically capture the sensing behavior, the electrical response of the sensors under gas exposure is simulated using detailed microscopic geometric data of the hetero-aggregates, allowing for accurate scaling of the simulation from grain-level to device-level behavior. Ultimately, the simulation framework acts as a cost function to identify optimal hetero-aggregate configurations and link them back to specific synthesis parameters, providing a predictive tool for designing high-performance SMOX gas sensors.

Content

Highly integrated light-excited gas sensors on LED microchips using flame spray pyrolysis

Dr Xiao-Xue Wang; Prof. Dr. Hua-Yao Li; Dr. Nicolae Bârsan; Prof. Dr. Lutz Mädler PhD

Abstract

The performance of semiconducting metal oxide (SMOX) gas sensors is largely dictated by the properties of their nanostructured sensing layers. Conventional fabrication methods, such as physical and chemical vapour deposition, sol-gel processes, and screen-printing, often face limitations including low deposition rates, poor scalability and the need for post-deposition heat treatment.

Flame spray pyrolysis (FSP) offers a transformative alternative. As a single-step aerosol-based process, FSP enables the direct deposition of highly crystalline, super-porous nanomaterials onto substrates with exceptional uniformity and reproducibility, and provides precise control over particle size, porosity, and layer thickness.

Here, we present a highly integrated dual-function device combining luminescence and gas sensing on a single microchip fabricated by FSP. The device comprises a transparent sapphire substrate with an LED layer and a gas-sensing layer on opposite sides. The sensing layer, activated by the LED, detects various gases at near room temperature with minimal power consumption. FSP enables the one-step deposition of ~10 nm nanoparticles with ~97% porosity and a specific surface area of ~80 m^2/g. This super-porous structure enhances both light penetration and gas response. By eliminating the need for post-deposition heat treatment, high-temperature interference with the LED emission layer is avoided. During deposition, a sample holder is positioned at a specific height above the nozzle, and circulating cooling water maintains the substrate at ~120 °C, enabling compatibility with various substrate materials. Additionally, FSP supports diverse material choices, including SMOX and metal sulphides, broadening the wavelength range of applicable LEDs.

Content

Synthesis of Si-Nanoparticles in a Radio Frequency Plasma Reactor (RF-Plasma)

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Abstract

Si-based anodes represent one of the next generation Li-ion battery (LIB) materials with a high potential, especially regarding their electrochemical storage capacity since silicon has a much higher capacity than currently used graphite-based anodes (theoretical capacity 4200 mAh/g vs. 372 mAh/g). Additionally, because of the high abundance of Si on earth, there are no issues regarding availability and sustainability. However, due to massive volume changes of up to 300 % during charging and discharging, Si is subject to great mechanical stress, which can lead to mechanical failure. Using nanostructured silicon material is favorable to prevent cracking and can thus result in enhanced electrochemical performance and long-term stability. For these reasons, there is a huge interest in the synthesis of Si nanoparticles (Si-NP).

A promising method for producing nanoscale silicon particles is the vaporization and recondensation of microcrystalline silicon powder in a high-temperature plasma, i.e. a radio frequency plasma (RF-plasma). The plasma reveals a high energy input and high temperatures (up to 10,000 K), which enables the vaporization of commercially available micropowders. Silicon (sieve size distribution: +45 mesh 0 % / -45 mesh 100 %) is aerosolized using a vibration powder feeder and vaporized in a TEK-15 nano plasma reactor (TEKNA, Canada). Crystalline silicon nanoparticles with a broad particle size distribution and a specific surface area of 28 m2/g corresponding to a BET diameter dBET of 93 nm can be obtained. The process technology enables a production rate of more than 30 g/h and can be increased by further process optimizations. Electrochemical measurements show that these materials exhibit promising properties as anode material in lithium-ion batteries.

Thermal conductivity of nanoparticle mixtures produced in sprouted bed

Subash Reddy Kolan
Otto von Guericke University

Abstract

Nanoparticles, due to strong cohesive forces like Van der Waals forces, naturally exist as homo-aggregates, where particles of the same type cluster together. For mixing nanoparticles of different materials, these homo-aggregates need to be broken down to individual nanoparticles or smaller aggregates. However, once dispersed, these nanoparticles tend to reaggregate because of cohesive forces. During this reaggregation, particles of different types attach to each other, forming hetero-aggregates, which are valuable for creating hetero-aggregates with unique properties. The ProCell spouted bed is an effective apparatus for producing hetero-aggregates of nanoparticles, as its high inlet velocity promotes the disintegration of larger homo-aggregates, while the design of the expansion chamber ensures a low superficial air velocity. This setup minimizes the loss of smaller particles through elutriation, thus optimizing the formation of hetero-aggregates.
In addition to particle mixing, the thermal conductivity of the resulting hetero-aggregates is an important factor, particularly in electronics industry. When nanoparticles of differing, thermal conductivities are combined in a gaseous medium, the effective thermal conductivity of the aggregate system changes in a moderate and smooth manner as the fraction of the more conductive component increases. This study focuses on analysing the thermal conductivity of hetero-aggregates produced within the ProCell 5 spouted bed. By evaluating how the thermal conductivity of the hetero-aggregates correlates with the homogeneity of the mixture, we aim to better understand the impact of particle composition and distribution on the thermal properties of nanoparticle aggregates.

Content

Reorganization behaviour of nanoparticle agglomerates under stress and strain: using machine lerning to gain insights from force spectroscopy data

Linus Hartz
FH Münster

Abstract

Porous films made from nanoparticle agglomerates are critical components in a variety of emerging technologies, including gas sensors, electrochemical energy storage, nanocomposites, and pharmaceutical formulations. These films are composed of hierarchically structured agglomerates. The agglomerates are formed by the clustering of sintered primary particles into aggregates. Physical forces hold the aggregates together to form larger assemblies called agglomerates. The internal reorganization of these structures under mechanical stress or strain has a significant impact on performance characteristics such as conductivity, adhesion, or mechanical integrity.
It is essential to understand how these nanoparticle agglomerates deform, separate, or reorganize under stress to predict and optimize their behavior in real-world applications. Chain-like nanoparticle agglomerates (CLA) unfold, deform, and rupture depending on environmental conditions such as humidity, surface chemistry, topology, or solvent environment. This behavior occurs in multiple stages, as previous studies have clearly demonstrated. The first stage is initial deformation, involving rolling and sliding of aggregates. This is followed by elastic deformation, and eventually plastic deformation, which leads to breakage. However, a predictive framework linking structural parameters and environmental conditions to mechanical behavior has not yet been established.
We investigate this using atomic force microscopy (AFM)-based force spectroscopy to directly probe the force–distance behavior of CLAs under defined environmental conditions. The analysis reveals a range of reorganization phenomena—such as sliding, twisting, rolling, and detachment—which manifest as distinct features in the measured force–distance curves. Distributions of detachment forces at constant humidity exhibit multimodal structures when measuring conditions are kept constant. However, with changing humidity, the multimodality vanishes. It is clear that there is no systematic dependence of the detachment force of

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aggregate-aggregate contacts on relative humidity. This raises the question if feature combinations in the force-distance curves can definitively indicate relative humidity.
To gain deeper understanding in this, data-driven methods are explored to extract latent features from the measured force curves. Classical machine learning models trained on engineered features achieved moderate predictive accuracy (~61–67%) in classifying the humidity condition under which a force curve was recorded. Convolutional neural networks (CNNs) trained directly on plotted force–distance data achieved significantly higher accuracy (up to 93%), demonstrating that deep learning models can identify subtle yet informative curve patterns that escape manual feature extraction.
We are currently training generative adversarial networks (GANs) to understand which structural elements in the force curves correspond to physical mechanisms of agglomerate reorganisation. These models identify latent parameters that define the characteristic shape of force curves associated with specific environmental conditions. The long-term goal is to decode the relationship between environmental parameters, particle-level structure, and mechanical unfolding processes.
This work contributes toward establishing a comprehensive predictive framework for the mechanical behavior of nanoparticle agglomerates. The insights gained may inform the tailored design of functional films and powder-based materials in fields ranging from additive manufacturing to targeted drug delivery.

Lagrangian simulation of hetero-agglomeration in turbulent pipe flows for the formation of solid-state battery cathode materials

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Abstract

The process of hetero-agglomeration, whereby multiple micron or submicron particle systems comprising different materials are brought together, has been shown to result in the formation of novel and remarkable properties in the resulting mixture. In the context of battery technology, hetero-agglomeration can be employed to create all-solid-state battery (ASSB) cathode materials comprising lithium iron phosphate (LiFePO_4, LFP) as active material, lithium halide (Li_3 InCl_6, LIC) as solid electrolyte and carbon black (CB) as conductive additive which promise an improved safety and an increased energy density of the battery cell [1]. The degree of homogeneity and the number of hetero-contacts formed between particles of different materials exert a significant influence on the resulting properties. Consequently, processes that enable such mixing and agglomeration – also with a view to industrial scalability – are imperative.
In previous works, a jet-based direct mixing gas phase hetero-agglomeration process was proposed and analysed numerically [2] and experimentally [3]. In this process, two or more powders of different materials are first deagglomerated and dispersed into the gas phase by rotating brush dispersers. Thereafter, the aerosols are amalgamated within a designated mixing zone, wherein particles have the potential to undergo hetero-contact formation. The numerical analysis indicated potential avenues for enhancing the initial setup, such as the elimination of a secondary carrier gas and the reduction of the agglomeration volume [2]. The modified process furthermore encompasses a turbulent pipe flow subsequent to the mixing zone, in which the materials are subjected to further mixing and agglomeration as a result of turbulence-induced collisions. Whilst previous studies have exclusively focused on binary particle systems and exemplary materials, the present study concentrates on the three-component hetero-agglomeration of ASSB cathode materials. Therefore, numerical

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simulations of the turbulent pipe flow hetero-agglomeration process are performed in order to analyse the mixing and agglomeration of the starting materials for the formation of the desired hetero-agglomerates.

It is evident that, due to the submicron size and the large number of the primary particles of the starting materials, widely utilised simulation approaches, such as discrete element method (DEM) simulations, are no longer runtime efficiently applicable for the overall process. Consequently, a suitable simulation framework was developed and presented in [4]. In this approach, a mesh-free representation of the flow field is obtained by providing curve-fitted functions derived from multiple DNS data sets [5] of turbulent pipe flows. The aforementioned functions provide, inter alia, the root-mean-square (RMS) values of the velocity fluctuations. The flow field for the actual shear Reynolds number of the pipe flow under consideration is obtained by means of a quadratic interpolation. An improved discrete random walk model [6] is utilised for Lagrangian particle tracking in the context of inhomogeneous turbulence and facilitates the modelling of dispersion and deposition, even in instances where the particles and agglomerates have very small Stokes numbers. The Saffman lift force of particles and agglomerates in the turbulent boundary layer is also considered in the form proposed in [7]. As the particles are tracked uncorrelated to each other, the agglomeration is modelled in the framework of a Lagrangian agglomerate structure approach, as primarily proposed in [8]. In the present study, the collision probability and the respective relative velocities of two particles/agglomerates are obtained through the semi-empirical model proposed in [9], which encompasses the turbulent shear and acceleration-induced collisions of bidisperse particles, also in the viscous subrange of the smallest turbulent eddies. This model also considers the effect of an increased collision probability due to a local preferential concentration between turbulent eddies, when the Stokes number is close to one. Furthermore, hydrodynamic interactions of colliding particles are of importance, as small CB aggregates may follow the streamlines of the larger LFP or LIC particles, without attaching. This effect is also taken into account through the introduction of an analytical approximation of the collision efficiency, as presented in [10], which also considers the effect of van der Waals attraction. In the event of a collision between particles, the collision velocity is compared with a critical velocity. If the relative velocities fall below this threshold, a collision will result in sticking. The critical velocity is calculated for each particle type pair by performing 1-D DEM simulations of particle-particle collisions within the framework of a Newton scheme initial to each simulation. It is important to note that each agglomerate is tracked as a whole (i.e. particle-like), where the corresponding diameter is

given by the radius of gyration. The internal structure of the agglomerates is only relevant in the event of collision.

The hetero-agglomeration of the three starting materials is simulated for different pipe radii under process conditions which have been examined experimentally previously [3]. The key process conditions are the particle mass flow and the gas volume flow. It is imperative that these conditions facilitate sufficient breakup and de-agglomeration of the primary particles, while concurrently maintaining a high particle concentration, thereby favouring re-agglomeration with heterogeneous particles. The particle size distribution of the primary LIC and LFP particles has been measured at the end of the brush disperser by means of laser light diffraction. Furthermore, the initial carbon black aggregates have been reconstructed from scanning electron microscopy images.

The numerical analysis demonstrates the manner in which the interplay between mixing, agglomeration and deposition on the pipe wall influences the resultant hetero-agglomeration and that the results strongly depend on the pipe radius. The differences in hetero-agglomeration can be attributed to an interplay of the following phenomena:

-The selection of smaller pipe radii whilst maintaining constant volume flow results in increased turbulence, which consequently enhances the probability of particle collisions. This phenomenon can be attributed to the increased accelerative collision mechanism, which is a consequence of higher fluctuation velocities and smaller turbulent (Kolmogorov) time scales. When the Stokes numbers are close to one for small pipe radii, the collision probability is further increased through local particle preferential concentration between the eddies.

-In the case of smaller pipe radii, the collision of LFP and LIC particles frequently does not result in agglomeration, as the collision velocity is higher than the respective critical velocity. While the agglomeration of the small CB aggregates on the other materials is favoured by the increased collision velocity, the rebounding of LFP and LIC compensates the influence of the increased collision probability on the agglomeration.

-More hetero-contacts than homo-contacts are observed in any simulation case and a pipe radius-dependent optimum has been identified. This is due to the disparity in surface energies and densities between LIC and LFP. When the turbulence induced collision

velocities are high in the case of very small pipe radii, the homo-agglomeration of LIC can be almost entirely prevented, while a large number of collisions between LFP and LIC particles still result in agglomeration.

-The deposition of particles and agglomerates is strongly increased when smaller pipe radii are selected. This phenomenon can be attributed to the increased effect of turbophoresis, which is a consequence of larger fluctuation velocities and smaller time scales. While the agglomeration is more evident in smaller pipes (at sufficiently low collision velocities), these agglomerates are deposited more rapidly. It is therefore possible to ascertain an optimum pipe diameter and length at which the agglomerates reach a desired size or possess a specific composition.

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Metal release and Nanoparticle formation in single droplet combustion

Jan Derk Groeneveld; Prof. Dr. Lutz Mädler PhD

Abstract

Flame spray pyrolysis (FSP) is versatile method capable of producing multi-component nanomaterials, able to combine seamlessly almost all elements in the periodic table. To gain a deeper understanding of the fundamental mechanisms that govern nanoparticle formation in FSP, single-droplet experiments have been employed – the smallest unit of the FSP process. For tracking metal release and the nanoparticle formation, high-speed imaging and flame emission spectroscopy were combined. This method allows for temporally resolved tracking metal release into the combustion. Combined with highspeed imaging, this approach provides unprecedented insights into the dynamics of precursor combustion and the nanoparticle formation. The analysis of various metal precursors revealed nanoparticle formation through a gas-to-particle process, identifying µ-explosions as the major mechanism for metal release and nanoparticle formation during combustion. The metal release was observed to be highly precursor-dependent, with some metal precursors only released via µ-explosions, while other, evaporable metal precursors were also released before the µ-explosion. The combustion of multi-metal precursors, Cu-naphthenate, with varied iron sources (evaporable ferrocene/rel. involatile Fe-naphthenate) showed simultaneous release of metals into the combustion via µ-explosions (Fe/Cu-naphthenate) and a release of iron before the µ-explosion and with copper during the µ-explosion with ferrocene. Resulting in a homogeneous product and a slightly heterogeneous product, respectively. These observations could explain the unique capability of FSP to produce both homogenous heterogenous nanoparticles. These insights enhance the understanding of FSP processes facilitating tailored nanoparticle production.

Content

Particle synthesis in confined microreactors

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Abstract

Single droplet experiments have been designed to study specifically the reaction and nanoparticles formation from metal-precursor-laden droplets that combust in air or pure oxygen. With the support of these experiments, physicochemical mechanisms in nanoparticle producing spray flames can be deduced. The single burning micro-droplets themselves can be viewed as spatially confined microreactors, with numerous mechanistic pathways involved in attaining the final product morphology.
In this work, the burning droplets with diameters D are confined in microfluidic channels, consisting of two parallel walls, separated by a specific gap distance H. This novel arrangement of burning micro-droplets within a microreactor allows precise control and fundamental investigation of droplet combustion dynamics. Various confined reactors with different gap sizes were set up and tested with burning solvent mixture droplets.
To study the influence of the confinement on burning phenomena, droplets containing a mixture of ethanol and xylene are combusted in different confined reactors with varying gap sizes. Both the flame dynamics and the histories of the droplets were analyzed as a function of the H/D ratio using high-speed imaging. Studying the burning dynamics shows that the flame sizes change with increasing confinement, leading to different temporal histories of the flame-to-droplet ratio. Using two synchronized high-speed cameras reveals that the proximity of the droplets to the wall and their interactions with it affect the burning dynamics and change the flame-to-droplet ratio.
These multiphase microflow systems can help increase the understanding of nanoparticle synthesis by using confined reactors to control gas flow reaction fronts and extract space and time resolved analyses.

Content

Controlled Synthesis of Copper Sulfide Nanoparticles in Oxygen-deficient Conditions using Flame Spray Pyrolysis (FSP) and its Potential Application.

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Abstract

Although the synthesis of metal sulfides using the FSP setup involves challenging process conditions, eight metal sulfides (MnS, CoS, Cu_2S, ZnS, Ag_2S, In_2S_3, SnS, Bi_2S_3) were successfully synthesized by precisely controlling the oxygen supply to the flame and ensuring an adequate metal-to-sulfur ratio. However, the fundamental understanding of the metal sulfide formation mechanism in the gas phase and the impact of process parameters on the particle characteristics are largely unknown. The objective of this study is to investigate the influence of different process parameters like fuel-to-oxygen ratio, precursor flow rate, co-flow rate, and different metal-to-sulfur ratios on flame spray pyrolysis (FSP) synthesized metal sulfide particle properties. The particle size increases with increasing the dispersion oxygen flow and copper sulfide is obtained only when the fuel-to-oxygen ratio is equal to or higher than 1.5. The temperature of the flame increases with increasing precursor flow rate and copper sulfide is obtained lower than or equal to 5 ml min-1 precursor flow rate while particles become contaminated above 5 ml min-1 feed rate. Co-flow rate above 100 l min-1 is necessary to cool down the aerosol stream before it is deposited on the filter. Pure copper sulfide phase is produced when sulfur is more than 5 times in molar ratio compared to Cu in the liquid solution and particle size decreases with increasing sulfur concentration. This research will contribute to a better understanding of the fundamental formation process of metal sulfide and in optimizing synthesis parameters for in situ coating, doping, mixing, and fictionalizing the metal sulfide nanoparticle in a gas-phase reducing environment.

Content

Formulation and development of graded stationary phase materials by Spray Printing

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Abstract

In this study, we present the formulation and development of stationary phase materials with property gradients by mixing nanosuspensions of different particle sizes via Spray Printing. The obtention of different functional properties of porous substrates is achieved by adjusting the mixing ratio of the nanosuspensions, the solid content in the droplets, the drying conditions and the height of the printed structure. A detailed analysis on the textural properties of the graded stationary phases is performed via high-resolution gas adsorption. Moreover, the optimal mixing ratio of binary mixtures for printing ideal structures with property gradients is investigated and analysed.

Comprehensive investigation of the SpraySyn 2.0 burner: characterization of particles, droplets and flame fluctuations using optical in situ diagnostics

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Abstract

Spray Flame Synthesis (SFS) is a promising technique for the production of metal-oxide nanoparticles. However, the processes involved in the synthesis and their effect on particle formation are not yet fully understood. Within the DFG priority program SPP 1980, the standardized SpraySyn burner 2.0 was developed, allowing for widespread and extensive investigations of the processes involved in SFS with multiple diagnostics on a common burner. In this study, comprehensive investigations of flame characteristics and flame stability, droplet size and particle formation using optical in situ diagnostics are presented. As material systems, iron-oxide (FexOy) and titania nanoparticles are investigated.
For the investigation of flame fluctuations, flame luminosity imaging using a high-speed camera is employed, allowing to assess relevant time scales and frequencies of the pulsation. Wide-angle light scattering (WALS) is used to determine droplet sizes in various heights above the burner (HAB). The particles produced by the SFS are analyzed using laser-induced incandescence (LII) and WALS. The WALS-results for various HABs show a slight increase in the particle size, with a dependence on the precursor concentration and the material produced. Simultaneous measurements of WALS and flame luminosity allow to investigate the correlation of flame pulsations and particle size. Using LII, the particle diameter is obtained from evaluating the thermal radiation signal of laser heated particles. Moreover, the spatially resolved particle mass concentration can be obtained using imaging LII, allowing to determine the height resolved relative particle mass and thus the main particle formation zone in SFS.
The work was supported by the German Research Foundation (DFG) - 375857474.

Synthesis and Reactive Laser Sintering of Copper Oxide and Alumina Nanoparticles for Production of Copper-aluminum Delafossite

Jeldrik Schulte; Dr. Martin A. Schroer; Prof. Markus Winterer
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Abstract

CuAlO2 is a transparent conducting oxide (TCO) with p-type conductivity. TCOs are widely used in photovoltaics and optoelectronic devices like flat panel displays or solid-state lighting. However, the performance of p-type TCOs in such devices is still significantly lower compared to n-type TCOs.
Chemical vapor synthesis (CVS) enables the synthesis of nanocrystalline metal ox-ide particles of high crystallinity, chemical purity, and narrow size distribution in the small nanometer regime.
Resonant laser sintering of nanoscaled metal oxide precursor powders is a versatile and innovative processing method to produce novel materials and functional com-ponents. When using photons with energies larger than the band gap of the green body material, fast and resonant heating is achieved with laser systems of rather low total power. This extremely fast and local heating as well as the subsequent cooling affect the sintering process and allow a fine control of the resulting (micro-) struc-tures.
In this contribution, we demonstrate the potential of reactive laser sintering of na-noscaled green bodies by investigating the processing and reactive sintering of CVS generated CuxO and Al2O3 nanoparticles via resonant laser sintering to gener-ate CuAlO2. Employing nanocrystalline powders as starting materials, p-type TCO-based devices with improved performance are expected.

Acknowledgements:
Funded by the Deutsche Forschungsgemeinschaft (DFG, German Research Foun-dation): SCHR 1753/1 & WI 981/19-1 (project nr: 531185908) and INST 20876/395-1 FUGG

Content

Photo-polymerization using flame-made quantum dots for stable epoxy coatings

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O Nanotech Solutions (CEO)

Abstract

Photo-polymerization is at the foundation of many industries such as dentistry, coating and stereolithography 3D printing. However, the organic cationic photo-initiators currently used are toxic, expensive, and difficult to tune with respect to the wavelength of light required to trigger polymerization reactions. For example, current stereolithography 3D printing resins are unstable under sunlight. Here [1], we demonstrate that cheaper and non-toxic titania quantum dots made via the scalable flame spray pyrolysis technology can photo-polymerize epoxy when exposed to UVC (not present in sunlight on Earth), while being insensitive to UVA (present in sunlight on Earth) leading to resins that are photo-stable during end use. We use NMR and FTIR to demonstrate that photo-polymerization is catalyzed under UVC but not UVA, and nanoindentation to monitor the mechanical stability of epoxy films during post-polymerization UVA exposure. This approach allows precise control over the wavelengths of light under which photo-polymerization can and cannot occur.

[1] Riad, et al., Ind. Chem. Mater., 2024, 2, 644

Charge transfer in particle-wall collisions

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TU Clausthal

Abstract

In particle-wall collisions, considerable amounts of charge are exchanged in addition to momentum and matter. This is a consequence of different surface potentials. Charge transfer even occurs between surfaces of the same material due to local inequalities, such as lattice planes, surface curvatures and adsorbates. This contribution shows how this charge transfer can be measured and how it can be used to characterize and separate nanoparticles and micrometer particles.
In the field of nanoparticles, low-pressure impaction combined with charge measurements can be used to obtain information about mechanical properties such as elastic and plastic deformation and the fragmentation of nanoparticles. In particular, critical velocity for bouncing, yield pressure and coefficient of restitution were determined for Ag and Au nanoparticles and compared to MD simulations.
In the micrometer particle range, contact with an impaction surface can be used to charge powders, which is known as triboelectric charging. By applying an electric field to the plate, the charge state of the particles can even be specifically adjusted (forced triboelectric charging). This approach allows powder mixtures to be separated according to one or more target components in the field of recycling. Very good results have already been achieved for the enrichment of lithium aluminate powder from the processing of lithium-ion batteries.

Content

In situ X-ray scattering of nanoparticle formation in Chemical Vapor Synthesis

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Abstract

Chemical vapor synthesis (CVS) is a scalable gas phase synthesis method to produce highly crystalline nanoparticles (NPs). Structural particle characteristics such as size, size distribution, agglomeration and crystallinity are controlled by the CVS process parameters, especially the time-temperature T(t). Control of particle characteristics enables the tailoring of properties of materials generated from these NPs. Usually, synthesized NPs are collected and characterized ex situ to determine the parametric influence. This leads to a loss of information regarding transient states during CVS and in the product due to artifacts during the collection process. Therefore, reliable information about the processes in CVS and related particle characteristics is only accessible via in situ probing. The key challenge here is a highly dilute gas phase as object of investigation with a number density of atoms 5 orders of magnitude lower than in corresponding bulk systems. However, modern synchrotron radiation facilities make such in situ experiments possible through their brilliant X-ray beams and detectors of high sensitivity. A novel mobile CVS reactor is designed for these experiments enabling control of the T(t) profile to produce tailored NPs [1]. We present results on simultaneous in situ small and wide-angle X-ray scattering experiments to probe size, morphology and microstructure of NPs during CVS.

[1] M. A. Schroer, A. Levish, Y. Yildizlar, M. Stepponat, and M. Winterer, A versatile chemical vapor synthesis reactor for in situ x-ray scattering and spectroscopy, Rev. Sci. Instr. 93 (2022) 113706, 11pp.; doi.org/10.1063/5.0122461

Prediction of flow regimes for flexible stalky particles in four-way coupled CFD-DEM simulations

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Abstract

Pneumatic transport is widely used for handling bulk materials, particularly powdery and coarse granular materials. It is also suitable for transporting biomass in stalk form, given its low density, which allows for conveying at relatively low fluid velocities. However, transporting biomass with elongated and flexible particles presents significant challenges [1]. The influence of particle shape on pressure losses and flow conditions is not well understood, and current design guidelines are inadequate. Further research is needed to address key issues, including drag force prediction and mass flow fluctuations to avoid over-dimensioning of pipelines to avert clogging, which leads to higher acquisition and operational costs.

Computational Fluid Dynamics (CFD) is utilized for the analysis and resolution of problems pertaining to fluid flows. In comparison, the Discrete Element Method (DEM) is employed to model particle movement in relation to contact forces and drag forces for instance, and representation of resulting local void fractions. The coupling of CFD-DEM can more accurately comprehend the complexities of particle-laden flows, enhancing the precision of predicting fluid-driven particle movement and therefore the accuracy of pressure drop predictions, aligning more robust with experimental data [2]. To examine the bending behavior of stalky and flexible particles, such as hay and straw, the bonded-particle method is employed as part of the DEM [3], with calibration of contact and bending parameters based on experiments involving single particles and particle systems of ideal and synthetic materials, such as e.g. cylindrical solid rubber cords.

In addition to the necessity for systems of pneumatic conveying to look at energy losses due to particle friction and impacts, the behavior of flexible particles in terms of bending, swinging and damping is also relevant [4]. It has been demonstrated that, owing to the increased energy dissipation, particles tend to sink to the bottom and accumulate as part of a particle-fluid flow. This phenomenon results in a transition from a fully suspended state to a strained

Content

or plugged flow leading to an increase in pressure drop. Additionally, stalky particles can interlock and clump together under the aforementioned conditions whereby forces on the particles increase depending on the exact shape and the orientation of the formed structure. The precise configuration of the formed structure is however pivotal in determining the predominance of particular forces. These may be area-based forces, such as drag, or volume-based forces, such as gravitation, which act in different directions. Consequently, the momentum exchange between solid and fluid phase is complex to predict. Moreover, the effective particle size often increases for clumped particles during flow resulting in possible unstable conveying or even pipe clogging.
This underscores the necessity of a valid parametrization of bending, swinging and damping behavior [4] in addition to frictional parameters [5], due to the influence of the relation between area- and volume-based forces on growth of clumped structures. Therefore, the bonded particle model has to be calibrated over experiments precisely as there is a substantial influence on the flow behavior. Based on this the resulting pressure losses over the superficial gas velocities are specified in relation to flexibility of the particles, and the particle movement and flow regimes are analyzed. This is intended to facilitate the design of pneumatic conveying systems that are significantly more sustainable.

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[3] D.O. Potyondy, P.A. Cundall, A bonded-particle model for rock, International Journal of Rock Mechanics and Mining Sciences. 41 (2004) 1329–1364.
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[5] C. Coetzee, Calibration of the discrete element method: Strategies for spherical and non-spherical particles, Powder Technol. 364 (2020) 851–878.

Evaluating the fibrillation level of a powder blend for fibrillation process improvement

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Granutools

Abstract

Dry-coating processes are new methods for electrode production that aim to replace the techniques using slurries. In such new techniques, the material is handled in powder form and directly ‘‘spread’’ on a current collector. However, compared to a slurry, powder material is challenging to handle due to poor flowability and has a complex rheology. Having good powder behaviours that match the requirement of a production line for battery production is challenging.

Polytetrafluoroethylene (PTFE) is a popular binder for dry processes. Its plasticizing properties are used to produce a thin film from a dry powder that is directly coated on a current collector to produce an electrode. This thin film is obtained by adding the PTFE to the active material (and the conductive additive) and then fibrillating the blend. During this process, the long chains of the PTFE are deagglomerated to produce long fibrils that form an entangled web with the active material, giving the plastic behaviour of the film. The level of fibrillation of the powder material highly influences the behaviour of the thin film and the final performance of the battery. During fibrillation, parameters such as mixing time, temperature and shear rate influence the level of fibrillation of the blend. In such a case, quantifying the degree of fibrillation according to these parameters is of huge interest for fibrillation process improvement.

In this work, we present how fibrillation can be measured and quantified with an improved tapped density method (GranuPack, GranuTools). Powder blends made of Lithium Iron Phosphate (LFP), PTFE, and carbon black were fibrillated under different conditions. They exhibit significant differences in bulk density, packing dynamics and flowability, allowing a differentiation and a quality control of the fibrillated blends. With this adequate method, the effect of different parameters such as temperature, shear rate or time on fibrillation can be investigated to adjust the best conditions for optimal fibrillation, improving the battery quality.

Content

Development of semiochemical-loaded microparticles by spray congealing for sustainable plant protection

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Abstract

Since insecticides are toxic even in small amounts and usually harm not only the target organisms but also other living organisms, the search for other plant protection strategies in agriculture is essential. One promising alternative to insecticides is the use of plant-insect or insect-insect communication molecules (semiochemicals) that can be used to manipulate insect behavior. Most products using this approach rely on synthetic polymers.
The key challenge with volatile semiochemicals is achieving optimal release rates, as improper concentrations reduce effectiveness. To overcome this problem and at the same time produce an eco-friendly product, we incorporate selected semiochemicals into microparticles where they are homogeneously dispersed in a matrix material. The material used is hydrogenated vegetable oil, which is biocompatible and a renewable resource. This strategy protects volatile substances and enables sustained release kinetics.
We produce these microparticles using an energy efficient and solvent free lab-scale spray congealing process. The goal is to produce particles that can be easily applied in agriculture as non-dusting powders/granules and don’t need to be retrieved, as the empty particles biodegrade in soil. Therefore, the spray congealing process was optimized to generate particles with a median diameter (d50) of around 300 µm. The influence of the process parameters atomization pressure, spray temperature and formulation mass flow on the particle size distribution was investigated. Reducing the spray temperature and pressure when increasing the mass flow rate result in larger particles. The largest particles (d50 = 296µm) were prepared using a spray temperature of 76.5 °C, an atomization pressure of 1.5 bar and a mass flow of 0.396 kg min-1.

Content

Formation of a stable SEI in Lithium-Ion-Batteries (LIB) via adding vinylene carbonate (VC) in anode suspensions

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Abstract

To enhance the longevity of LIBs formation of a solid electrolyte interphase (SEI) is crucial for stable operation. VC is often added to the electrolyte as an additive to facilitate the SEI formation. The advantages of VC include an improved battery life cycle, fast SEI formation, and a high charge transfer resistance. However, VC is electrochemically unstable on the surface of high-voltage cathode active materials (CAM) leading to undesired side reactions. Thus, innovations are imperative for stable SEI in high-voltage CAM, e.g. the formation of an artificial SEI or introducing VC during anode manufacturing. The production of an anode slurry consists of high-shear mixing active materials, binders, and conductive additives in a solvent in specific ratios to achieve uniform dispersion and prevent agglomeration. In this processing, VC can be easily added, so that less VC is required, as the VC added to the electrolyte is not quantitatively polymerized during the formation process and side reactions on the cathode are avoided due to spatial separation.
This study investigates the targeted treatment of electrode materials prior to cell assembly to apply a SEI, offering an alternative for achieving customized SEI and preventing migration of VC to the cathode. To achieve this target different concentrations of VC are added in the dispersion processes. The processability and interactions at the interface, e.g. hydrogen bond, electrostatic interactions and Van der Waals forces, of the suspensions are assessed based on viscosity, particle size distribution, Hansen solubility parameters , and pH. The influence of the coating and drying parameters of the electrode sheets and the concentration of VC on the resulting electrochemical properties are investigated in coin cells.

Content

Influence of hydrothermal treatment on the foaming behaviour and porosity of PLGA/RHA composites

Marco Pelaez[2], Kilian Händler[1], José Manuel Vázquez, Ignacio García Casas[3], Belen García Jarana[3], Pablo García-Trinanes[3]

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[2] Westsächsische Hochschule Zwickau
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Abstract

The development and modification of biodegradable polymers has gained increasing scientific and technological attention due to the growing demand for sustainable and environmentally friendly materials (Álvarez *et al.*, 2020; de Macedo Rooweder Lima, 2024). Derived from renewable sources, biodegradable polymers such as PLGA emerging as viable alternatives to conventional plastics, with wide-ranging applications, particularly in biomedical engineering, where controlled porosity and biocompatibility are critical for tissue engineering and drug delivery systems (Valor *et al.*, 2021; Zhou, Tian and Zhang, 2024)

This study investigates the combined effect of hydrothermal (HT) pre-treatment and supercritical CO_2 foaming (foaming) on PLGA 75:25 composites containing rice husk ash (RHA) as a bio based inorganic filler. Cylindrical samples were prepared with three RHA concentrations (10%, 25%, and 33% w/w) and subjected to either foaming alone or HT followed by foaming. Expansion factors were calculated based on volume changes, characteristics such as particle size, morphology, and pore size distribution were analyzed to understand their role in the pore formation.

The inclusion of RHA aimed to introduce nucleation sites that promote microstructural changes during foaming. Experimental results showed that all RHA containing samples exhibited higher expansion factors compared to blank PLGA, with the HT + foaming treatment producing the most evident improvements. SEM images confirmed surface modifications and more uniform pore openings, with tomography results already offering very revealing insights.

These findings highlight the potential of RHA as an effective nucleative agent while emphasizing the role of hydrothermal treatment in enhancing pore nucleation and distribution. The sinergy between bio based inorganic fillers and, solvent free processing strategies provides a sustainable route to design the microstructure of biodegradable polymers composites for advanced functional applications.

Content

Effect of mineral additive structure on PLGA expansion during supercritical CO_2 foaming

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Abstract

PLGA is a biodegradable polymer widely used for porous scaffold applications (Carrascosa *et al.*, 2024). Two inorganic additives Wollastonite (WLS) and Bentonite (BT) were combined with PLGA to investigate their influence on CO_2 foaming process. The additives were chosen based on their different morphologies. WLS has a needle-like shape and BT has the form of lamellar plates(Hadal *et al.*, 2004).
Samples were produced with two additive concentrations (⅓ and ¼ wt% additive) and treated with supercritical CO_2 foaming at 45 °C, 200 bar and 30 min, with and without a prior hydrothermal pretreatment (HT) at 100 °C, 25 bar and 10 min. The expansion factor was calculated from initial and final volumes using a cylindrical model.
Wollastonite showed the highest expansion, especially under HT+Foaming conditions, reaching up to 10.6. Bentonite samples expanded more moderately and consistently (with an average of 4.3), with occasional outliers in Foaming only cases. Without the HT both additives showed lower overall expansion.
These results suggest that wollastonite promotes more effective bubble nucleation due to its fibrous structure particularly after hydrothermal treatment(Faba *et al.*, 2024). Bentonite provides more controlled, stable expansion but limits overall porosity(Piperopoulos *et al.*, 2022).

Content

Leveraging Powder Characterisation to Predict Dissolution and Dispersion in Pharmaceutical Formulations

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Abstract

The dissolution and dispersion of pharmaceutical powders are critical parameters influencing drug efficacy and manufacturing efficiency. Predicting these behaviours from fundamental powder properties is challenging, especially as lumps and aggregates often form during powder-solvent mixing, compromising product quality. This study addresses these issues by experimentally characterising pharmaceutical excipients to identify key material attributes governing dissolution performance.

Advanced techniques are used to conduct quantitative measurements of particle size distribution, flowability, and cohesion. These data offer insights into how particle-level attributes influence dissolution stages, including wetting, sinking, swelling, agglomeration, and dispersion. Systematic comparisons between powder properties and controlled dissolution experiments reveal which critical quality attributes dictate dissolution rate and extent.

Building on these findings, a data-driven computational framework is developed using deep learning algorithms. Training predictive models on high-quality characterisation and dissolution data reduce reliance on time-consuming experimental trials. It streamlines formulation development, accelerates process scale-up, and improves the reliability of predicting product performance under varied conditions.

The outcomes highlight the importance of linking material properties to dissolution mechanisms. This enables targeted formulation optimisation, consistent product quality, and enhanced scalability. More importantly, it ensures reliable prediction of dissolution profiles early in the design phase, supporting the development of safer, more effective pharmaceutical products.

Content

Detection and Machine Learning Modeling of Spray Nozzle Blockage as a Process Anomaly in Fluidized Bed Spray Granulation Processes for Quality Assurance

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Dr.-Ing. Swantje Pietsch-Braune; Prof. Dr. Stefan Heinrich
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Abstract

Fluidized bed spray processes are widely used in food, pharmaceutical and chemical industry for several decades, but the complex interactions between solid, liquid, and gaseous phases still pose challenges for process control and optimization. One of the most frequent anomalies is spray nozzle blockage, which disrupts homogeneous liquid distribution. The blockages often arise from uneven liquid flow, particle deposits, or equipment wear, necessitating precise detection methods.

This project focuses on developing a systematic, data-driven framework for detecting spray nozzle blockages during fluidized bed granulation. To address this challenge, various sensor technologies are evaluated, including flow sensors to monitor liquid supply rates and optical sensors such as light detectors to detect spray patterns and particle build up. These measurements are integrated into an innovative knowledge module that combines sensor data with machine learning models to enable accurate detection of nozzle blockages.

Rigorous data pre-processing ensures the reliability and robustness of the detection system, providing actionable recommendations to mitigate the effects of such anomalies.

By focusing on this specific and critical anomaly, the framework aims to improve process reliability, prevent production interruptions as well as waste of material and energy, and ensure consistent product quality by predicting anomalies in fluidized bed spray granulation processes.

Content

Inverse identification of the mechanical parameters and coupling with improved internal structural analysis of frozen particle fluid systems

Amirali Khosrozadeh; Dr.-Ing. Swantje Pietsch-Braune; Prof. Dr. Stefan Heinrich
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Abstract

The Discrete Element Method (DEM) with the Bonded Particle Model (BPM) is widely used for simulating frozen solid particulate systems particularly those exhibiting brittle and quasi-brittle failure mechanisms. BPM employs virtual bonds to replicate elastic deformation, crack initiation, and propagation. Accurate calibration of DEM parameters is essential to replicate experimental mechanical behavior. However, traditional trial-and-error approaches for parameter calibration are time-consuming and computationally expensive.

This study introduces a robust inverse method for calibrating DEM-BPM parameters to match simulated and experimental macro-behavior during compression tests. Design of Experiment (DOE) methodology, particularly the Taguchi method, is employed on parameters to identify the most influential BPM microparameters. The identified parameters are then adjusted using an inverse calibration approach to ensure consistency with experimental observations of the compression test.

The structure of the DEM geometry is derived from micro-CT scans, which provide high-resolution 3D images of the internal structure of frozen particle fluid systems with different volumetric ice content and particles. Therefore, a specialized compression test apparatus has been developed for in-situ testing within the micro-CT device, allowing simultaneous acquisition of mechanical and structural deformation data.

Results demonstrate that combining the Taguchi method and inverse calibration produces a reliable DEM-BPM model that replicates experimental force-displacement behavior. This integrated methodology provides a robust tool for understanding the mechanical behavior of particle-fluid systems and opens new pathways for parameter calibration in DEM-BPM simulations of complex materials.

Content

CFD-DEM simulation of structure formation during the drying of fuel cell electrodes

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TU Braunschweig

Abstract

Fuel cells are a sustainable technology for generating electrical energy and serve as an alternative to batteries in mobility applications. A key component of the fuel cell is the membrane electrode assembly (MEA), whose microstructure determines the performance and lifetime of the entire system, offering significant potential for optimization. A critical process affecting the electrode's microstructural properties is drying, which influences porosity, pore radius distribution, and the distribution and cross-linking of the catalytic material. To better understand and predict how drying and formulation parameters affect the electrode structure, simulations combining computational fluid dynamics (CFD) and the discrete element method (DEM) can be applied. This study presents a CFD-DEM simulation model for detailed analysis of liquid-particle interactions during drying. To accurately reproduce the film formation process, the simulation environment's numerical parameters were optimized, and the representation of the catalyst material's fractal aggregates was improved. Furthermore, sensitivity analyses were conducted to assess the impact of different contact and interaction models on catalyst layer formation. The simulation environment enables analysis of how catalyst material parameters (e.g., fractal dimension, pre-factor, particle and aggregate sizes) influence the formed layer’s structure (e.g., thickness, porosity, pore size). Results were then compared with experimental data to evaluate the model's applicability.

Content

Investigating the Effect of Mechanical Property and Realistic Shape of mRNA-LNP during Spray Drying via CFD-DEM Coupling

Jiqian Guo PhD; Konstantinos Giannis; Silas Wolf; Dr. rer. nat. Jan Henrik Finke; Prof. Dr.-Ing. Carsten Schilde

Abstract

The stability and efficacy of mRNA vaccines are highly influenced by their sensitivity to environmental factors, which requires innovative strategies for long-term storage. Drying processes, such as spray drying and lyophilization, have shown advantages in enhancing the stability of mRNA-lipid nanoparticle (mRNA-LNP) formulations. However, during drying, mRNA-LNPs are exposed to thermal and mechanical stresses, such as shear forces, which can impair their structural integrity and functionality. For better understanding the drying process of mRNA-LNP formulations, a spray-drying simulation model is developed by coupling computational fluid dynamics (CFD) with the discrete element method (DEM). Regarding that lipid nanoparticles may possess unique mechanical properties due to the internal multilamellar or unilamellar structure, such as a hard core-soft shell structure, a novel contact model was implemented to improve simulation accuracy and reliability [1]. Mechanical characterization test and drying process coupled CFD-DEM simulations with this contact model are conducted for the sensitivity analysis of mechanical properties. Additionally, the influence of non-spherical particle models could be considered using particle models generated through a generative adversarial network (GAN), which provides the possibility for a detailed analysis of shape-dependent structural dynamics. This study provides insights into the drying-based structure formation of mRNA-LNP formulations of vaccine, contributing to the optimization of drying process and the development of more reliable vaccine storage solutions.

Content

Elucidating cluster and orientation tendencies of nanocellulose particles in slot die coating using CFD-DEM simulations

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Abstract

The shift to sustainable paper packaging is a major trend in the food industry. Due to the poor barrier properties of mono paper, extra protective films are needed to inhibit contaminant migration. Nanocellulose, effective against grease, oil and oxygen, is a promising candidate. The coating process impacts barrier quality, making it essential to understand nanocellulose behavior during slot-die coating. The CFD-DEM method, combining computational fluid dynamics (CFD) with discrete element modeling (DEM), provides detailed insights into particle-fluid interactions.
CFD-DEM simulations were performed with nanocellulose modeled as a sphero-cylindrical particle with an aspect ratio of 20 in water, using coarse graining to reduce the computational effort. The suspension was modelled as a Casson Fluid. Both one-way coupling and two-way coupling between the particle and the fluid phase were employed. The study focusses on the particle orientation during the application step and the interaction between the slot-die flow field and nanocellulose's agglomeration tendencies. A distance criterion was established to evaluate clustering under varying conditions, identifying trends in particle proximity. Additionally, two different angles were defined to characterize the orientation of nanocellulose during the coating process.
Results show that changing flow fields have minimal impact on the orientation of nanocellulose, which consistently aligns parallel to the substrate and the axis along the flow direction. First results indicate that the locations where agglomeration is likely to occur are not random, with the upstream area and locations close to the wall being particularly prone. This trend was consistent for varying flow conditions and coupling methods.

Content

DEM Simulation of Impact Charge on Particles by Particle Impacts Across Different Chute Lengths

Mehran Javadi MA[1]; Dr.-Ing. Annett Wollmann PhD[2]; Prof. Dr. Alfred. P Weber[2]
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Abstract

Triboelectric charging, a phenomenon where materials acquire charge through frictional contact, is pivotal in numerous industrial applications, particularly in material separation and recycling. This study introduces a novel method known as Forced Triboelectric Charging (FTC). In FTC, a high voltage is applied to a surface (chute), and particles are introduced to interact with the wall, enabling the charge of the target material to be selectively neutralized while other materials acquire charge. Following tribocharging process, an electric field (separator) is employed to recover and separate the neutral particles from the charged ones. Understanding of the charge mechanisms and the factors that influence particle charging behavior is crucial for optimizing recovery processes. Experimental results from the FTC process revealed that chute length plays a significant role in the charging behavior. Specifically, increasing the chute length improves the specific charge on the particles, but charge saturation occurs from a certain length. To further understand this phenomenon, in this study, Discrete Element Method (DEM) simulations are employed to model triboelectric charging of particles on the chute. Simulations are conducted for chute lengths of 200, 600, 900, 1200, and 1800 mm, replicating experimental conditions. The model focuses on charge transfer during single and repeated particle impacts, to find the correlation between chute length, number of impacts, and cumulative particle charge. These simulation results are then compared with experimental data to validate and refine the understanding of the process.

Content

Modeling Swelling Behavior in Nano-Silicon Coated Graphite Anodes Using Discrete Element Method: Insights into Particle-Level Interactions and Irreversible Volume Changes

Kashfia Mahin
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Abstract

Nano-silicon-coated graphite anodes offer a promising solution for improving lithium-ion batteries by combining the high energy storage potential of silicon nanoparticles with graphite's structural stability. While nano-silicon exhibits reduced volumetric expansion (~20–30%) compared to bulk silicon (~300%), its dynamic behavior during cycling remains complex. This study applies the Discrete Element Method (DEM) using the Thornton-Ning model to simulate swelling and shrinking behavior, analyzing key parameters such as the carbon binder domain (CBD), friction coefficient, Thornton-Ning yield ratio, Young's modulus, and restitution coefficient to validate experimental data.

Lithiation is modeled based on diffusion-controlled expansion, where lithium diffusion follows Fick's law, scaling with t1/2 , and a diffusion coefficient of 10(e−14) to 10(e−10) cm2/s. The particle radius growth is expressed as a function of initial radius, volumetric expansion coefficient, and lithiation depth. Using Taylor series approximation, the formulation indicates proportional particle radius increase with time for small expansion ratios, while nonlinear effects are captured with additional terms.

The simulations reveal that higher silicon content increases swelling, while a 10% rise in friction coefficient reduces particle dislocation by ~5%. The Thornton-Ning yield ratio and Young's modulus significantly influence deformation, with stiffness values above 10 GPa reducing structural changes.

This study bridges computational modeling with experimental observations, offering quantitative insights into microstructural and diffusion-driven behavior in Si/C composite anodes. The findings establish a foundation for optimizing electrode designs to enhance durability and cycling stability.

Modelling of Drying and Solid Formation of a Levitating Saline Droplet

Benjamin Mignot[1]; Dr. Tariq Mahmud PhD; Prof. Dr. Peter Heggs; Prof. Dr. Mojtaba Ghadiri PhD; Prof. Dr. Kevin Roberts
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Abstract

The drying kinetics and morphology of dried particles have applications in many processes, from spray drying to spray coating or fluidized bed drying. Population balance modelling (PBM) is a deterministic modelling approach that allow to account for the nucleation and growth of crystals in a drying droplet.
This study aims to model the solid formation of a levitating saline droplet by mathematical modelling of its evaporation (validated [1]) and crystallisation. Numerical solutions are obtained for the water evaporation, heat transfer, and internal solute (NaCl) diffusion. The number and size of crystals are predicted by PBM with both the discrete method and the method of moments [2].
Good agreement is found between the model predictions and experimental data from drying a saline droplet using an acoustic levitator for the change in droplet size over time. Using power law models for the crystal birth and growth rates, both the full discrete method and the method of moments produced similar results. However, such birth and growth parameters were seen to have a large influence on the predicted number and size of crystals at the end of evaporation.
This study demonstrates that the method of moments can accelerate predictions of final particle morphology, while emphasizing the necessity of precise nucleation and growth modelling.

A DEM-Based Ultrasonic Field Method for Particle Levitation

Dimitri Ivanov[1]; Marvin Röhl[1]; Trung Anh Nguyen[1]; Jan Raffel[2]; Dr. Christoph Lotz[2]; Prof. Dr.-Ing. Carsten Schilde[1]
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Abstract

The levitation of particles via ultrasonic fields holds substantial promise for a variety of applications, including particle transport, separation, and precise targeting. Although the finite element method (FEM) is often used to simulate such scenarios, its combination with the discrete element method (DEM) can become computationally demanding. In this work, we adopt the DEM approach to effectively capture particle behavior under ultrasonic excitation, thus avoiding the high computational load typically associated with FEM. The ultrasonic pressure field is represented by a piston radiator operating at a single ultrasonic frequency, and the force exerted on particles is determined using the negative gradient of the Gor'kov potential. To account for the amplitude of a single transducer, experimental pressure measurements across distance were employed for calibration, and the manufacturer's directivity data were used to model the reduction in power away from the center. An experimental setup based on Marzo et. al. was developed to validate the simulations, featuring two opposing 16×16 ultrasonic transducer arrays. These arrays generate a standing wave that enables the levitation of particles with varying densities by creating equilibrium regions where the particles remain suspended. Dynamic adjustments of the acoustic field at each time step target specific coordinates, allowing precise particle positioning. The outcomes highlight the promise of this fully DEM-based method for advanced particle handling, opening up new possibilities in engineering contexts where precise control over particles in dynamically shifting acoustic fields is crucial.

Content

New Opportunities for the Dynamic Simulation of Solids

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DyssolTEC GmbH

Abstract

Dynamic simulation of solids processes - Dyssol is of a growing interest in solids processing like in the chemical, ceramic and mining industries. The mathematical description and therefore the ability to predict product quality is becoming increasingly important, increasingly with the help of machine learning tools. But starting on a low data basis, the design of experiment (DOE) for the determination of optimal process parameters is a frequently used approach. With higher dimensions, the experimental effort increases dramatically, depending on the chosen method (e.g. full factorial, Box-Benke) resulting in a low level of variations per dimension, and therefore only linear and cubic relationships in the extracted model.
Model based DOEs can reduce the experimental effort, as a predefined physical or empirical set of models is iteratively adjusted after each experiment, to determine a good fit. The ”Parameter Estimation” module of Dyssol®Pro allows the optimization of unit parameters based on experimental data. This allows the model-based DOE approach utilizing Dyssol models for solids processes, and with this the chance for upscaling the process applying Dyssol. After determining the refined process model, a tie-in simulation with other process units allows optimizing complete process chains by applying the inbuild “Optimizer” module. The Dyssol-model-based DOE approach shows a solid method for reducing the experimental effort for daily DOE work.

Methodological Approach for Understanding Particle Transport. Case Study: Analyzing Influential Factors and Comparing Simulation Software

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Prof. Dr. Daniel Wiest[1]; Prof. Dr.-Ing. Henning Meyer[1]
[1] Technische Universität Berlin; [2] Volkswagen AG

Abstract

Recent research shows that the investigation of particle flows of small particles with diameters smaller than 10 micrometers has emerged as an important field of interest, especially for purposes in the industrial sector. Investigating the behavior, deposition and acting forces of individual particles has become particularly relevant. Understanding theses dynamics is crucial for setting up practical simulations in terms of particle transport drawn from industrial problems. Previous research has already identified numerous forces involved in particle transport e.g. (Sommerfeld, 2000), (Crown, 2005), (Löffler & Raasch, 1992) or (Crown, et al., 2011). However, not all of them have yet been implemented in practice for different reasons. In this case study, acting forces are examined theoretically using a component coming directly from the automotive industry. The examined component is a spout of a vehicle was chosen which is assembled within headlights. Due to its simple structure, it is conductive to analyze the particle-laden flow in different simulation software. The simulation setup for this component less complex, which saves time and reduces costs. The implementation of the examination processes within different simulation software is also analyzed. Simulating a particle-laden flow results in significant computational costs. It is necessary to identify and implement boundary conditions. All results serve as the basis for developing an appropriate simulation model and to create a methodological approach representing particle transport across different scales. The simulation results are validated by comparing them with appropriate experiments. Furthermore, a transfer function was derived to determine the filtering effect of an automotive component. This study combines theoretical background with a practical application.

Content

CFD-based investigation of agglomeration in spray drying with fines return

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[1] Otto-von-Guericke University Magdeburg;
[2] Łódź University of Technology

Abstract

Spray drying is a widely used technique to produce powders from liquid feeds, with applications across various industries. To enhance the efficiency of the process, undersized particles, fines, can be recovered from the exhaust drying air and returned to the drying chamber. This procedure aims to encourage the collision of fines and fresh feed droplets and the formation of larger agglomerates. While fines return is a standard practice in many industrial spray drying operations, this process is not yet understood in all of its details. This lack of understanding can limit the ability to fully optimize the fines incorporation during spray drying. The present study systematically investigates the effects of various operational conditions and geometrical variations on the fines incorporation rate and the particle size distribution of the powder product. To achieve this, computational fluid dynamics simulations equipped with advanced models accounting for key phenomena such as droplet drying, coalescence, and agglomeration are used as the primary tool. An assessment of the simulation's accuracy is made with the help of own experimental trials conducted on a technical-scale spray dryer.

Content

Correlation of 2D and 3D Particle Characteristics from Simulated Imaging Data

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Abstract

The translation of particle shape and size information from 2D into 3D has been a long-pursued endeavor of researchers in the field of particle technology. Necessarily, correlating 2D and 3D results is always severely limited, but differently so depending of the chosen approach: shape may be predicted accurately only for simple geometries; small amounts of data may not show a holistic picture; real particle geometries are difficult to come by through simulation.

In a recent publication (https://doi.org/10.1016/j.partic.2024.10.008), real particle geometries as determined with X-ray microtomography were used to simulate static and dynamic image analysis. The results were used to find correlations for some common questions, especially: how does the particle projection correlate with particle width and how particle sphericity (3D) is linked to 2D shape descriptors. Over 6000 particles were used for this study that comprise six different solids ranging wildly in particle shape from highly spherical to plate-like, to find general predictors of 3D particle properties, rather than finding correlations that excel in describing any particular bulk solids.

Correlations for the case of dynamic image analysis were determined by using a graphical approach of coloring markers in scatter plots according to several candidate parameters. This method proved fruitful in revealing hidden variables. We find that particle width and Wadell's sphericity is predicted well for compacted particles by a fixed relationship of different geometric shape parameters. Both correlations have been validated by dynamic image measurements of the bulk solids that have been used in this study, thereby knowing the 3D property distributions.

Content

GP-based Modeling for PSD Control of Emulsification Processes

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Abstract

Although there exist different modeling approaches for particulate systems, most of which are variants of population balance models, they are typically too complex and high-dimensional for control applications. Due to this reason, emulsifications are often controlled in an open-loop scheme. Applying closed-loop control schemes to emulsification processes promises an improvement of overall product quality and process efficiency.
This presentation shows a modeling approach centered around the use in closed-loop control schemes for the case study of an emulsification plant. The dominant dynamic behavior of the considered process is the particle breakage induced by the high shear mixer (HSM). Due to the inaccessibility of the HSM, the need for a physical description is bypassed by a data driven modeling approach. To reduce the model size and make it suitable for the use with obtainable datasets, the particle size distribution (PSD) is modeled by a combination of one-sided normal distributions. The dynamics of the hyperparameters are obtained using Gaussian process (GP) regression based on PSD measurements. The GP-based model design additionally provides a measure of uncertainty for the parameter propagation, which can be used to determine the uncertainty of the discrete PSD description using an unscented transformation. This additional information is of advantage for the control of particulate processes, where some amount of model and measurement uncertainty is expected.
This talk is based on the author's paper "GP-based Modeling for PSD Control of Emulsification Processes" presented at ICSTCC 2024 (Proc. pp. 477-482), adding new results for different use cases and substance mixes.
The work was supported by the German Research Foundation (DFG) – project number 504809428.

Dynamic Calibration of DEM-Simulations Using Rotating Drum Experiments

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Abstract

In the present work, a refined calibration method for discrete element method (DEM) simulations of granular materials is introduced, which are used to enable precise predictions of the process behaviour of granules. The key feature of the method is the use of a rotating drum to capture dynamic characteristics of the bulk material under standardized and repeatable conditions. Unlike in static observations, where the amount of available characteristics is limited, the dynamic approach provides a wide range of possible key parameters that can be evaluated and are closer to the actual behavior during operation. To fully exploit this potential, automated image analysis is used to extract key parameters like the dynamic angle of repose, the detachment and attachment points of the particle bed, as well as the general shape of the particle bed surface, ensuring high accuracy and repeatability. The Python code for this automated image analysis was developed in-house, allowing for customization to the specific needs of the drum experiment and can be downloaded from the GitHub page of the project. The extracted data serves as the basis for iterative adjustments of the simulation parameters, which are carried out until a predefined level of accuracy in the match between the experimentally obtained values and the corresponding values from the DEM simulation using ANSYS-Rocky is achieved.
Machine learning techniques such as neural networks in conjunction with genetic optimization algorithms are employed to guide the calibration process, saving computational time and determining optimal simulation parameters more efficiently than could be achieved through trial and error. The calibrated simulation models are initially validated by comparing the model predictions for simulations with varying parameters, such as different rotational speeds and fill levels, to experimental results that are performed with a GranuDrum instrument (Granutools, Belgium). This comparison shows that the proposed method

Content

produces reliable and consistent results for steel spheres under various operating conditions. The approach is further validated with experimental data obtained from planetary ball mills in collaboration with Fritsch GmbH. In this case, the conditions (such as speed and motion regimes) under which the calibration was performed differ significantly from those to which the simulation is applied. Nevertheless, it is shown that a sufficient agreement in the results can be achieved. Finally the method is applied to non-spherical particles. For this purpose, irregular shaped potassium chloride granules were used in drum experiments and additionally measured using a micro-CT system to obtain detailed information about the actual particle shape and geometry to be used for calibration. So far, it has been shown that the highly irregular geometry can be represented in the simulation programm ANSYS-Rocky. However, the computational time required to achieve a sufficiently large number of simulations currently poses a challenge. Further work in this area is planned for the future and will be discussed in the outlook.

Numerical Investigation of Inclined Plane Flow of Highly Deformable Particles Using a Calibration-less Bonded-Sphere Model

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ETH Zürich

Abstract

Dense granular media flowing down an inclined plane are frequently encountered in nature (e.g., landslides) and in industrial processing operations (e.g., discharging operations). Previous research concerning the physics of granular inclined plane flow has predominantly focused on rigid particles featuring a high Young's modulus, while the flow of deformable particles such as rubber and microgels that are commonly used in industry has gained significantly less attentions. Hence, this work aims to adresses this gap by modelling and elucidating the dynamics of inclined plane flows of highly deformable particles.
To this end, a calibration-less, bonded-sphere model based on the discrete element method (DEM) is developed and implemented. This models allows the simulatation of linearly elastic, highly deformable particles. Each particle is segmented into sub-spheres to capture its motion and deformation by resolving intra-particle and inter-particle interactions via virtual bonds and contacts between sub-spehres. The model is carefully validated using well-established single and multi-particle scenarios.
Subsequently, the newly developed DEM model is applied to simulate inclined plane flows of different angles of inclination and Young's moduli. Our simulations reveal that, unlike rigid particle flows in which the solid fraction and inertia number is uniform, flows of highly deformable particles exhibit an increased solid fraction and inertia number near the bottom plate owing to the increased pressures. Additionally, flows of deformable particles show an orientational ordering that is similar to the ordering in rigid, non-spherical particles, i.e., the particles are aligned at an angle with the flow direction.

Content

Discrete modeling of limestone particle calcination and carbonation under isothermal conditions

Enqi Liu PhD; Prof. Dr. Abdolreza Kharaghani[1]
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Abstract

Discrete modeling of pore evolution in limestone particle calcination and carbonation
Enqi Liu, Xiang Lu, Abdolreza Kharaghani
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Abstract
Building on our previous work on discrete modeling of pore evolution in a single limestone particle during calcination, this study extends the model to include both pore expansion due to CO_2 production (forward reaction) and pore shrinkage from CO_2 accumulation in pore walls (reverse reaction). These competing phenomena are modeled through local thermal energy balances, coupled with the transport of gas species. The local reaction rates for calcination and carbonation are determined by kinetic parameters, active surface area, and gas species partial pressure. The results show the evolution of solid grains and pore structures over time, accurately capturing local conversion profiles and pore size distributions. By accounting for both pore expansion and shrinkage, the model provides a realistic representation of structural changes. These findings are essential for developing accurate single-particle continuum models and contribute significantly to understanding transport in reactive porous media.
Acknowledgement
Funded by the Deutsche Forschungsgemeinschaft (DFG, German Research Foundation) – Project-ID 422037413 – TRR 287.

Content

Prediction of Moisture Uptake in Blister-Packed Solid Pharmaceutical Dosage Forms: Insights from a Case Study

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[1] Invite GmbH; [2] Bayer Consumer Care AG; [3] Bayer AG; [4] TU Dortmund University

Abstract

Blister packs represent one of the most prevalent forms of packaging utilized for solid pharmaceutical dosage forms. A principal objective of blister packaging is to preserve the product and thus ensure the quality of the medicine. For a considerable number of pharmaceutical products, water represents a factor that impairs stability and is consequently regarded as a critical attribute in this context. In this study, a modeling framework was developed with the objective of predicting the moisture uptake of pharmaceutical tablets.
A numerical approach to the interconnection of the diffusion processes of water was utilized for the prediction of moisture sorption in a blistered tablet. In the model, the water barrier properties and geometric information of the packaging material were combined with the sorption properties of the tablet. In order to ascertain the sorption kinetic and sorption isotherm of the tablet, a dynamic vapor sorption (DVS) analysis was conducted. Simultaneously, the prediction was validated by monitoring the mass increase of blistered tablets.
The higher relative humidity of the environment results in the ingress of moisture into the blister, which is subsequently sorbed to the tablet. The simulation was confirmed by the observed increase in tablet mass within the available time frame for the current experiment. This case study demonstrates the feasibility of forecasting the moisture uptake of blister-packed tablets through the presented numerical approach. As the modeling framework is founded upon parameters that can be determined comparatively fast, a reduction in time can be achieved in contrast to an experimental assessment. This allows for greater efficiency in addressing key questions associated with the selection and design of blister packs.

Content

DEM-aided risk reduction in a pharmaceutical powder bin blending process

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[3] Graz University of Technology

Abstract

In pharmaceutical tablet manufacturing, an evenly mixed powder is necessary to ensure the processability of the powder as well as the correct drug dosage per tablet. The design of powder blending processes still relies heavily on expensive trial runs. These costs can be reduced by guiding the process design with discrete element method (DEM) simulations.
In this work, the mixing dynamics of two active pharmaceutical ingredients (APIs) and an excipient component in a bin blender were modeled using the commercial DEM code XPS (InSilicoTrials). The key step in DEM models of powders is the calibration of the particle contact parameters which define the powder flow. The flow behavior of the three different powder components was studied experimentally using a dynamic angle of repose tester. Contact parameters for the three components were selected from a database to match the experimentally observed flow.
A simulation of the entire 25-minute blending process predicted an uneven distribution of the APIs along the rotation axis. Targeted sampling and high-performance liquid chromatography (HPLC) analysis in an experimental trial confirmed this trend. Different segregation mitigation scenarios were studied in the DEM model. The adoption of a baffle in the blending container improved the blending speed and the final blend homogeneity. Alternatively, a 90° rotation of the blending container before the final blending stage was able to drastically reduce the axial concentration variances without requiring an engineering effort.
This work highlights a state-of-the art DEM workflow for the study and design of multi-component powder mixing processes. The presented approach can identify process risks and evaluate mitigation strategies, thus reducing costly experimental trials.

Content

Modeling the entire filtration process from depth filtration to surface filtration with a multi-layer construction and an offset for dust cake growth

Dr. Qian Zhang
University of Wuppertal

Abstract

A new, clean filter medium for dust separation always begins with an initial phase in which dust separation can be described according to the principle of depth filtration in the given medium. If dust separation is carried out on the given filter medium for a sufficiently long time, a dust cake can form after a completed clogging phase. This is often a gradual process. Until now, there has been no coherent model for a holistic view of the entire filtration process. While the filtration kinetics of a depth filter can be investigated using various model approaches (dendrite growth model or fiber growth model) to describe particle separation in the clogging phase, it is not yet possible to transition to surface filtration by continuing the same model calculation.

This paper presents a new modeling approach with a multi-layer construction and an offset for the growth of dust cake, which enables the description of particle separation by using analytical models throughout the entire filtration process without a separate add-on model for the change between depth and surface filtration. The continuous evaluation of the locally resolved structural changes, i.e., in the individual sub-layers, as well as the associated changes in the separation efficiency of the single-collectors in the growing two-component filtering system provide insightful information about the filtration kinetics during the entire filtration process and about the process of dust cake formation. Both general results of the model calculation and targeted model calculations are presented.

Content

Comparing and contrasting calibration methods for granular materials

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Abstract

Accurate calibration of discrete particle method (DPM) simulations is essential for reliable virtual prototyping of granular materials. However, identifying DPM parameters that reproduce observed bulk behavior is challenging and ambiguous. To address this, we created datasets using virtual prototypes of characterisation machines using a synthetic granular material with known DEM parameters. Two datasets were used: shear-cell data alone and combined shear-cell and rotating-drum data.
We compared three calibration methods: Bayesian inference (GrainLearning), neural networks, and random forests. These methods differ in approach: GrainLearning seeks a single optimal parameter set by focusing on regions with low bulk response error, while neural networks and random forests create surrogate models that map the relationship between material parameters and bulk properties across the entire parameter space.
Results show all methods can identify parameter sets that approximate bulk behavior, but none deliver a unique solution. GrainLearning's performance depends on the parameter search range and improves with a constrained parameter space. The surrogate models' effectiveness depends on sampling strategy, though grid sampling introduces artifacts in random forest models. Using multiple characterisation machines improves calibration accuracy for all methods.
Key insights are:
(1) Refining sampling strategies for surrogate models enhances accuracy.
(2) Selecting appropriate characterisation machines reduces experimental effort.
(3) Hybrid calibration approaches improve efficiency.
These insights provide a guide to more reliable and computationally efficient calibration of granular materials and minimise the need for extensive experimentation

Content

Modeling the Mechanical Response of Halide Solid Electrolytes and Performance in All-Solid-State Batteries Using Discrete Element Method

Cerun Alex Varkey[1]; Kostas Giannis; Dr. Sebastian Melzig; Prof. Dr.-Ing. Carsten Schilde; Prof. Dr.-Ing. Sabrina Zellmer
[1] Fraunhofer IST

Abstract

Halide solid electrolytes stands as a strong choice for the next generation batteries considering its fast ionic conductivity, currently achieving conductivities higher than 2 mS cm□1. Application of pressure at different steps during the production and assembling of separator and catholyte components influences its performance inside a cell. This is influenced by the microstructural properties of the halide and cathode active material particles. The microstructural properties of the particles plays a crucial role in understanding the mechanical response of the components. Here, we propose a new contact model using Discrete Element Method (DEM) that calculates the normal forces considering the elasto-plastic behaviour [1] and by simultaneously contemplating all neighboring contacts of the contacting particles [2]. The particle structures of the solid-state separator and the catholyte components are simulated using the force response by the proposed contact model. The force response at the contact and its influence on the overall structure and surface of the components are evaluated. Furthermore, the mechanical ageing behaviour of the composite cathode structure is evaluated considering the changes in volume and elastic properties of the cathode active material during de-/ intercalation of Li+ ions. The generated microstructure of the halide separator and catholyte component is evaluated to determine the contacting paths between the particles and thereby calculating the ionic and electrical conductivities of the separator and cathode components. This approach enables the optimization of various pressures during both cell assembly and operation, identifying the appropriate processing pressures that improve the electrochemical properties of the components.

Entropy-based Powder Microstructure Analysis using Micro-X-ray Tomography

Frederik Dörr PhD
AstraZeneca

Abstract

The manufacturability of pharmaceutical materials is strongly influenced by bulk powder behavior, such as packing and lumping, which directly impacts performance during continuous direct compression (CDC). In this study, we present a novel application of micro-X-ray tomography (micro-XRT), a non-destructive 3D imaging technique, to provide high-resolution insights into the microstructure of pharmaceutical powders.
We employ kernel-based methods to transform 3D image data into local spatial density distributions, reducing the complexity of these datasets to a 2D representation. These distributions are characterized using established statistical metrics, enabling the quantitative comparison of powder microstructures across samples. Spatial entropy, derived from information theory, is introduced as a robust metric to quantify packing heterogeneity, capturing variations in local packing densities and distinguishing between homogeneous powders and those with large voids, air pockets, or dense agglomerates.
In cases where traditional segmentation techniques are impractical due to challenging primary particle attributes, this entropy-based characterization offers a simplified yet powerful alternative for assessing powder microstructure. By reducing 3D data complexity through automated workflows, this approach ensures reproducible and efficient analysis of routinely collected datasets.
Our findings highlight the effectiveness of spatial entropy as a quantitative descriptor of powder packing, providing valuable insights into structure-performance relationships critical for oral product development. This workflow enables rapid material screening for CDC compatibility, offering a transformative approach for routine material characterization in pharmaceutical development.

Content

In silico design space investigation of a novel tablet coating process using advanced coupled simulations

Dr Peter Böhling[1]; Dr Dalibor Jajcevic[1]; Frederik Detobel[2]; James Holman[2]; Laura Bower[3]; Matthew Metzger[3]; Prof. Johannes G. Khinast[4]
[1] Research Center Pharmaceutical Engineering GmbH; [2] GEA Process Engineering NV; [3] Merck Sharp & Dohme Corp; [4] TU Graz

Abstract

Continuous manufacturing is increasingly used in the pharmaceutical industry, as it promises to deliver better product quality while simultaneously increasing production flexibility. GEA developed the ConsiGma line(s) to be integrated into granular downstream processes. The specially developed tablet coater is unique in its design and process mechanics. It uses high rotation rates compared to traditional drum batch coaters.
Computational methods are increasingly used in the pharmaceutical industry as well and facilitate a deeper understanding of the process mechanics. The discrete element method (DEM) and computational fluid dynamics (CFD) are two methods that allow transition from empirical process design to a mechanistic understanding of the individual process units. This work used CFD-DEM simulations to increase the understanding of the ConsiGma® tablet coater through a multi-variant design study. The results are split into two categories: first, the tablet bed behavior (mechanics) will be investigated and second, the thermodynamically behavior will be studied.
The goal of this work is to present a comprehensive overview of the influence the varying process and material input parameters have on the process output responses. The simulations show how the drum load, rotation rate, and material properties all influence the tablet bed behavior. The same can be said that the drying air flow rate, spray rate, and inlet temperature can influence the coating quality which is tracked through the tablet temperature and wetness. Overall it could be shown that simulations allow for the full coating process to be recreated numerically which, in return, saves material and equipment time typically required for process development.

Content

Data is all you need? An experience report about the challenges of data preparation for machine learning-based analysis of food spray drying processes

Florian Kaltenecker; Prof. Dr.-Ing. Reinhard Kohlus; Prof. Dr. Christian Krupitzer
University of Hohenheim

Abstract

The process control of spray dryers is generally not considered critical, as there are only a few adjustable parameters. Nevertheless, the control behavior is highly complex and nonlinear. Therefore, conventional control strategies, especially PID controllers, often reach their limits for large-scale spray drying plants. This limitation arises from the non-ideal drying behavior of the products and the dynamic nature of the spray drying process. Particularly in poorly defined systems, such as during the scale-up of new equipment or product reformulation, drying behavior becomes difficult to predict accurately. As a result, extensive preliminary trials are often required. Digital twins of the process, based on suitable models and data, can provide valuable insights into the expected performance of the dryer.

Traditional modeling approaches in spray drying are based on physical and mathematical descriptions of the process. These range from simple balance-based models to advanced computational fluid dynamics (CFD) models that simulate energy and mass transfer on individual particle trajectories [1]. While these models are well-suited for capturing heat and mass transport phenomena, they often reach their limits when it comes to integrating physical-chemical product properties, complex product–process interactions, macroflows, and residence time distributions [2, 3].

Recently, the application of artificial intelligence, particularly machine learning (ML) for data analysis, has gained increasing importance in spray drying applications [4, 5], such as particle size prediction [6] or the combination of computer vision with machine learning for process analysis [7]. Such machine learning approaches share the commonality that they integrate a machine learning pipeline comprising data collection, data preprocessing, data

augmentation, feature engineering, algorithm selection, training, and analysis model development using the algorithms, as well as model evaluation. For data analysis using machine learning, the “Garbage in, garbage out” statement describes the relevance of data quality: The analysis results will only be usable if the input data is of high quality. At the same time, sufficient volumes of training data are required to ensure generalizability of the models. Consequently, for machine learning, much time is spent on data collection and pre-processing.

A review of the existing literature reveals that limited training data remains a frequent challenge in the field. In most cases, ML models for spray drying have been trained on fewer than 100 data instances [8-14]. Strategies to reduce the amount of data required for ML models, such as augmentation techniques [15] or cross-validation methods [16, 17], or the use of structured design of experiments (DoE) [9], can help mitigate this issue. However, these are still rarely applied in practice.

Moreover, the selection of ML algorithms applied in spray drying research remains narrow. Most studies employ artificial neural networks (ANNs) to predict product characteristics [8-17]. Alternative ML methods for modeling process parameters have only been explored in a few instances [6, 18, 19]. While existing studies suggest that ANNs often perform well [6], systematic comparisons with other algorithms are lacking and could support the identification of more robust or interpretable models.

Another limitation is the predominant focus on data from laboratory-scale spray dryers. Only a few studies have been conducted on a pilot scale [8, 10], even though transferring results from the lab to an industrial scale remains challenging due to scale-dependent phenomena, such as varying residence times.

Finally, many existing studies rely solely on naive, data-driven ML approaches. Even for processes like residual moisture prediction, where a solid understanding of the process in research exists, basic physical principles, such as mass balance considerations, have not been included in ML models to predict product moisture.

The present project aims to address several of these limitations. A central composite design is employed to systematically generate training data on a pilot-scale spray dryer. Subsequently, the data is augmented using SMOTER [20], Conditional Tabular Generative

Adversarial Networks (CTGAN) [21], and a multi-step machine learning framework, as described by [15], to obtain sufficient data for training machine learning models. The data is then used to train a selection of machine learning models, including k-nearest neighbor regressors, random forest regressors, and artificial neural networks. Furthermore, the effect of incorporating physics constraints into the loss function of the neural network on predicting residual moisture is investigated. The goal is to identify the most effective pipeline structure for predicting product parameters in pilot-scale spray drying. The application focuses on predicting residual moisture in spray-dried products. This simplified model serves as a foundational framework for developing a machine learning pipeline, which can be adapted for more complex predictive tasks.

In future work, the developed machine learning framework will be utilized to parameterize physical–mathematical models, e.g., a CFD simulation. The physical-mathematical models define the fundamental behavior of the system, enabling extrapolation based on the available data. This facilitates the use as a digital twin. Digital twins are virtual representations of physical objects, systems, or processes. They are used to simulate, monitor, and analyze real-world counterparts in real-time or near real-time, often by using data collected from sensors and other sources [22]. By combining physical–mathematical modeling with data-driven parameter estimation in a hybrid digital twin [23], the goal is to achieve more accurate predictions of both system dynamics and product characteristics.

In the poster presentation, we highlight the data preparation challenges for analyzing food spray drying processes using machine learning. It describes the workflow from systematic analysis of required data to controlled data collection and pre-processing techniques. Specifically, we highlight the obstacles to achieving high data quality and provide measures for improving data quality, focusing on data analytical tools and procedures that are most effective in achieving sufficient data quality and quantity for machine learning applications. As this is a recently launched research project, no research results were available at the time the abstract was submitted.

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Multi-Scale Modeling Approach for Solids Flowsheet Simulations: Rotary Kiln for Calcination Process of Zeolite Materials

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BASF SE

Abstract

The aim of flowsheet models is the simulation of production process chains, that are represented by connected process models of single unit operations. These flowsheet models can be used for design, optimization, or control purposes of chemical processes. Due to the high complexity of such processes, the flowsheet approach relies on a simplified representation of the single process steps using models of reduced complexity, mostly with respect to the spatial dimensions. For solids processes, however, the apparatus geometry has a great impact on the process itself and the material behavior, showing the necessity of incorporating apparatus specific phenomena in simplified process models.
In this work, a framework is presented that aims to link data obtained from particle-level simulations with flowsheet models. This approach not only allows for the incorporation of apparatus geometry in the simplified models but also enhances their predictive power by providing additional information at the particle level. Depending on the process, this can be data like residence times or holdup of the materials in different regions of the apparatus, collision rates and intensities, uptake of a liquid spray or the number of particles taking part in chemical reactions. Since the data typically depend on process conditions, it is useful to conduct a simulation study instead of a single simulation at the particle level. The resulting data can then be used to derive multidimensional correlations. With these correlations as input, the influence of process parameter on the solids behavior can be depicted with the simplified models. As an example, a rotary kiln calcination process for the production of zeolite materials is used in this study.

Content

Robust and efficient parameter calibration for DEM simulations of shear tests based on Bayesian inference

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Abstract

The predictive accuracy of Discrete Element Method (DEM) simulations depends on the precise calibration of its input parameters, such as particle elastic and dissipative/frictional properties. Traditional calibration techniques often rely on trial-and-error or optimization approaches, which can be computationally expensive and prone to subjective bias. In this study, we explore the application of Bayesian inference for the robust calibration of DEM parameters in the context of simulated vs. experimental shear cell tests. The Bayesian framework provides a probabilistic perspective, enabling parameter estimation as well as quantification of uncertainty associated with the calibrated values. We apply a methodology leveraging the open-source package GrainLearning [1], specifically designed for machine learning-driven parameter estimation of DEM simulations, coupled with an extended version of the open-source MFIX [2] package for DEM. By using advanced sampling techniques, it accelerates convergence by identifying the most influential parameters and focussing on regions of high likelihood. The role of particle dissipative/frictional parameters on the macroscopic mechanical response, i.e. stress-strain behavior, in shear cell tests is discussed.
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Exploring the Effects of Interparticle Forces on the Rheological Properties of Micro Particle Suspensions Using Unresolved Coupled CFD-DEM Simulations

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Abstract

In micro-particle suspensions, a variety of forces govern the interactions among particles. Accurately modeling these systems requires not only accounting for fluid-particle contact forces but also incorporating both normal and tangential contacts between the particles themselves. However, standard approaches such as coupled CFD-DEM simulations often overlook certain critical forces, hindering a faithful representation of the suspensions' rheological behavior. In this work, we examine how different interparticle forces impact the flow properties. Our results confirm that a comprehensive approach - including contact, static friction, rolling friction, drag, rotational drag, and lubrication forces - is vital for capturing the rheological characteristics of micro-particle suspensions. Contact and static friction address the direct particle contact, while rolling friction is essential to represent the influence of non-spherical particle shapes. Drag and rotational drag govern the acceleration and deceleration of translational and rotational motion; despite being commonly reserved for resolved simulations, rotational drag is shown to be crucial for accurately predicting suspension viscosity. Lubrication forces, reflecting the fluid's displacement between particles, additionally foster energy dissipation within the suspension. Moreover, electrostatic, steric, and van der Waals interactions must be included for colloidal suspensions. Altogether, our simulations incorporating these forces align well with both theoretical models and experimental observations across a wide range of solid volume fractions.

Content

A Multi-Sensor and Co-Kriging Approach: Toward Innovative Meta-Modeling of Ceramic Powder Milling

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Abstract

The efficiency of ceramic powder milling in ball mills is strongly influenced by several key parameters, including the filling rate, ball-to-powder ratio, milling speed, and the intrinsic properties of the powder. In this study, we examine how these parameters impact the evolution of particle size distribution during the milling process.

To capture the complexity of the phenomenon, we implemented a multi-sensor approach that combines high-speed imaging, acoustic emission sensors, accelerometers, and periodic granulometric sampling. High-speed imaging enabled us to monitor the dynamic milling phenomena with fine temporal resolution, revealing the intricate interactions between the milling media and the powder. Additionally, the analysis of acoustic and vibrational signals allowed us to extract energyrelated parameters, providing valuable insights into the impact dynamics and particle displacement mechanisms within the mill.

The experimental data have been collected with the aim of integrating them into a co-kriging model, which may help predict particle size distribution as a function of processing conditions, initial powder characteristics (both morphological and mechanical), and real-time sensor outputs. Although preliminary investigations are promising, further work is needed to validate the predictive capability of this approach and to better understand the relationships between operating parameters and milling efficiency.

Our findings suggest that the multi-sensor methodology, when combined with advanced statistical techniques, holds potential for enhancing our understanding of milling phenomena.

Content

In particular, the observed correlations between energy parameters derived from acoustic and vibrational signals and the evolution of particle size distribution point toward promising avenues for improved online process monitoring and optimization. Ultimately, this integrated approach may contribute to better process control and efficiency in the industrial processing of ceramic powders.

Enhancing CFD efficiency in spray drying through presampled and time-averaged airflows for accelerated simulations

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Abstract

Spray drying is a widely used process in industries such as food, pharmaceuticals, and detergents due to its ability to rapidly convert liquid feed into dry powder with precise control over particle characteristics [1]. However, the process is inherently complex, involving turbulent, threedimensional airflow fluctuations and dynamic interactions between particles and hot air. These interactions create spatial and temporal gradients in temperature and humidity that significantly affect drying behavior, leading to challenges in scale-up and accurate modeling. Particle phenomena such as agglomeration, crust formation, and wall deposition further contribute to the complexity [2, 3, 4]. Computational Fluid Dynamics (CFD) is the most common approach for simulating spray drying, as it resolves local-scale interactions governed by fluid mechanics [5]. However, fully transient CFD simulations are computationally intensive due to the need for fine mesh resolution, complex discrete phase modeling (DPM), and long simulation times to capture unsteady flow behavior [6]. These demands limit their practical use in rapid process optimization or screening of feed formulations, especially in industrial settings with limited computational resources and time constraints [7]. To address this limitation, a hybrid CFD workflow for simulating a co-current spray dryer that significantly reduces computational time while maintaining accuracy is examined. The method involves first performing a simplified transient simulation to capture the dynamic air behavior, from which time-averaged velocity fields are extracted. This "frozen" airflow field is then reused for steady-state particle tracking, avoiding the need to resolve transient gas-phase dynamics repeatedly. Building on this baseline, a two-factor parametric study is conducted in with the feed-water mass fraction and the inlet-air temperature are systematically swept. The resulting database of particle temperature, moisture content, mean diameter, and other related quality metrics is used to fit a loworder polynomial surrogate (linear or quadratic regression) that maps the two inputs to the key outputs. The surrogate is then coupled with a cost function which is used to predict the operating conditions that best satisfy the targeted product specifications without further CFD runs.

Results show that this hybrid approach yields predictions comparable to a fully transient model in terms of particle behaviour and dryer performance, while reducing simulation time from approximately three days to five minutes using pre-sampled airflow fields. Using a surrogate model trained on the CFD-generated dataset, computation time is further reduced

to under 10 seconds per prediction. The surrogate, based on low-order polynomial regression, accurately captures the relationship between feed-water mass fraction and inlet-air temperature to key product metrics (e.g., particle temperature, moisture content, mean diameter), achieving an R^2 above 0.97 and an relative error of 2% across all outputs within the studied parameter space.

In conclusion, the proposed hybrid CFD method—coupled with a surrogate-based optimization framework—offers a practical and computationally efficient alternative to fully transient spray dryer simulations. By drastically reducing simulation times from days to minutes (or even seconds with the surrogate)

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Digital twin development for high shear granulation processes

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Abstract

High Shear Granulation (HSG) is an important unit operation in the production processes of products such as pharmaceuticals and enzymes, enabling the transformation of powders into dense granules for improved physical attributes. Despite its industrial significance, predicting product properties from HSG processes remains challenging. Current mechanistic approaches combine Discrete Element Methods (DEM) for particle flow dynamics with Population Balance Modelling (PBM) for granule property prediction [1]. However, their numerical solutions are computationally expensive, particularly when applied to industrial scale processes. This limits the applicability of such DEM-PBM models as Digital Twins (DT) for real-time prediction.

This work proposes a novel surrogate modelling (SM) framework to accelerate DEM simulations through advanced machine learning (ML) techniques, enabling efficient integration with PBM for computationally tractable DTs. The research methodology includes developing and validating a DEM-PBM model on a pilot-scale batch HSG, which will also be used to generate a comprehensive dataset for training data-driven SMs. The ML architecture will build upon recent advances in relational bias invariance techniques and model scale to handle the complexity of particle interactions in high shear dense granular systems.

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Systems and Tools to Establish a Digital Shadow for Particle Generation Processes

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Abstract

Particles can be generated with precise features. Therefore, many different process routes like sol-gel processing, precipitation processes or flame-spray pyrolysis are used. In order to comply with FAIR principles, a digital description of these generation methods is required. Important features of particles are gained by characterization methods. This information is combined to create the concept of the digital shadow.
A digital shadow is a digital representation of an actual object that utilizes an automated one-way data transfer between the current state of a physical object and a digital representation. To achieve the digital shadow of particles, we use an electronic laboratory notebook (ELN) and semantic technologies to describe particle generation based on an application ontology within the frame of a core ontology called PMDco (Projekt Material Digital core ontology). The data are used to feed an ontologization routine where the terms and their quantities are processed with the help of microservices to deliver a semantical data set for this particular experiment.
To map these complex process routes as a digital workflow, we start to use a workflow management system (WMS). Because the ELN stores every process, measurement or characterization as single experiments, it is not easy to keep overview about all experiments within one workflow. In contrast, a WMS enables the tracking of all these experiments required to produce and characterize the resulting particles as a clear representation, but the most important features of a WMS are the capturing and presentation of interrelations between the steps and actions concerning the particle generation and characterization. With that, the real particles can be mapped as one digital object or as a digital shadow.

Content

Accelerating Innovative Pathways for Materials Synthesis: Towards Autonomous High-Throughput Nanoparticle Spray-Flame Synthesis

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Abstract

Spray-flame synthesis (SFS) is a scalable technique for producing functional nano-particles, particularly multielement oxides. However, optimizing synthesis pathways is challenging due to the complex parameter space and time-intensive reactor recon-figuration. To overcome these limitations, we propose a high-throughput SFS approach integrated with in situ and inline diagnostics for autonomous process optimization. This enables precise control over key product composition and characteristics such as particle size, morphology, and crystal phase while systematically exploring compositional landscapes for advanced materials in catalysis, energy storage, and beyond.

High-throughput techniques are particularly advantageous for SFS, as they facilitate both material evaluation and scalable production, ensuring sufficient material for real-world testing. Additionally, the high-temperature process stabilizes compositionally complex oxides and enables access to material phases that are difficult to achieve by other methods.

In this work, we develop and implement an autonomous high-throughput SFS platform. Our system integrates real-time reactor monitoring for process stability and control, along with direct inline diagnostics via automated nanoparticle sampling and rapid property characterization. A machine-learning-driven planning algorithm processes materials data provides immediate feedback, correlates in situ observations with properties, and iteratively refines reactor conditions for optimized synthesis. This approach significantly accelerates materials synthesis pathways and paves the way for an autonomous lab, expediting the transition of novel materials into innovative technologies.

Content