# Doped Aluminas Silica-aluminas Mixed metal oxides Hydrotalcites



## **Sasol Chemicals**



## About us

We at Sasol Chemicals innovate for a better world and deliver long-term value to our customers, communities and society.

Our broad portfolio of high-value products plays an integral role in the creation of numerous solutions that benefit the lives of millions of people.

Thousands of companies around the world leverage our technology, world-class facilities, expertise and collaborative approach to tackle their challenges.

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## **1.** Alumina production process

Sasol Inorganics produces high- and ultra high-purity alumina primarily through synthetic aluminum alkoxide processing routes. The alumina is produced either as co-product with synthetic linear alcohols (Ziegler method) or directly from aluminum metal (on-purpose route).

Several production steps must be completed to produce the different alumina-based products. In the first step, an aqueous intermediate (alumina slurry) is produced, which is further tailored in the subsequent processing steps to obtain the various products sold on the market. These can be alumina hydrates, calcined aluminas and doped versions thereof.

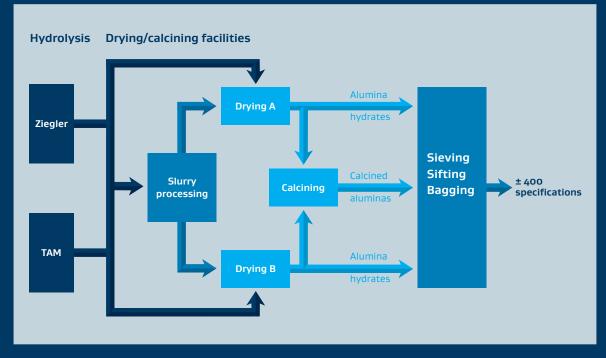


Figure 1: Schematic for the alumina manufacturing process

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# 2. Doped aluminas and mixed oxides

Sasol has manufactured high-purity aluminas and alumina hydrates with tailored physical properties for 50 years. The development of doped aluminas and mixed oxides led to a further diversification of our product portfolio with an even higher level of optimization for final use. The combination of dopants and doping technologies is another important step towards support materials with well-defined phase composition leading to improved properties like thermostability and acidity.

Silica-aluminas exhibit a higher degree of acidity than pure aluminas. The hydrates are sold under the trade name **SIRAL** and the corresponding oxides as **SIRALOX.** 

Mg/Al mixed hydroxides and oxides exhibiting more basic surface sites are indispensable support materials for diverse applications. These materials are sold as **PURAL MG** and **PURALOX MG**.

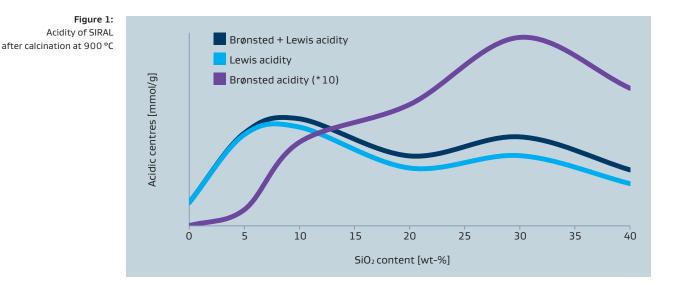
Besides the formation of mixed oxides, also the addition of other elements to the pure alumina, silica-alumina or mixed Al/Mg oxides and hydroxides is a facile way to further improve the properties of the corresponding starting materials for several applications. For example, lanthanum-doped aluminas exhibit a much higher thermostability in comparison to the non-doped counterparts.



#### 2.1 SIRAL/SIRALOX

The concentration and type of acidic sites (Lewis acid sites versus Brønsted acid sites) present in the materials strongly depends on Al<sub>2</sub>O<sub>3</sub>/SiO<sub>2</sub> ratio as it can be seen from figure 1. In general, the total acidity is governed by Lewis acid sites with weak and moderate strength. The highest concentration of Lewis-acidic sites is observed for **SIRAL 5** while the highest amount of Brønsted-acidic sites is found in **SIRAL 30** and **SIRAL 40**.

The more recently developed **SIRAL HPV** series exhibits a larger pore volume and a more homogeneous Si-Al distribution associated with a higher concentration of Brønsted acid sites, as it can be seen in figure 2 (results from Pyridine adsorption/desorption).



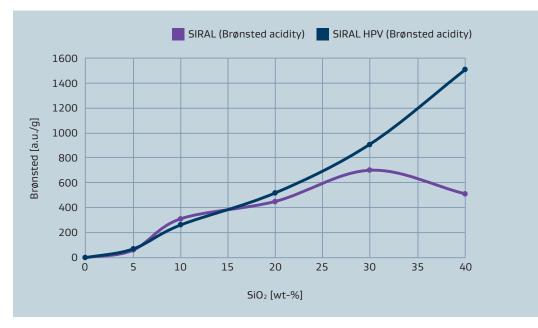


Figure 2: NH<sub>3</sub>-TPD of SIRAL 40 types after calcination at 900 °C

Besides differences in the chemical properties, also some physical characteristics like surface area are in marked contrast to pure aluminas as illustrated in figure 3.

As different applications also demand for different physical properties, these can be tailored by varying the base alumina.

The flexibility which is possible in case of the silica-aluminas is represented in figure 4. The different physical properties of **SIRALOX 5** depend on the corresponding starting alumina.

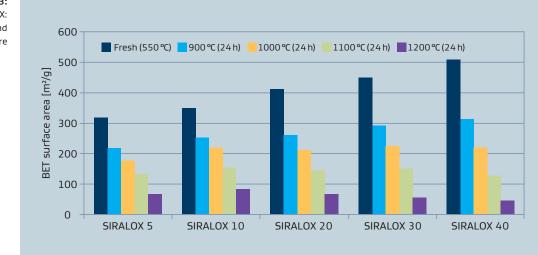


Figure 3: Surface area of SIRALOX: effect of silica content and temperature

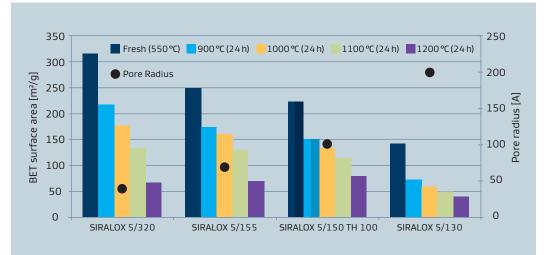


Figure 4: Characteristics of SIRALOX: effect of different starting aluminas

#### 2.2. PURAL MG/PURALOX MG

Sasol has adopted the alkoxide-route also for the production of high-purity Mg-Al mixed hydroxides and oxides.

Hydrotalcites (Layered Double Hydroxides, LDH) with general formula

 $Mg_{1-x} Al_x (OH)_2]^{x+} [CO_3^{2-}x_{/2} \cdot nH_2O]^{x-}$  are in general highly alkaline in nature and exhibit a pronounced affinity towards the adsorption of  $CO_2$ . These features can be adjusted by variation of the Mg/Al ratio and/or the addition of dopants, e.g.  $K_2CO_3$ . Moreover, different anions can be incorporated into the interlayer region of the LDH structure, thus leading to different basal spacings along with modified chemical and physical properties.

The calcined version **PURALOX MG 26** consists of the phase-pure spinel MgAl<sub>2</sub>O<sub>4</sub>. This oxide is another attractive catalyst support material for applications which require a lower degree of surface acidity than obtained in pure alumina. In addition, **PURALOX MG 26** exhibits a high surface area of up to 100 m<sup>2</sup>/g and high chemical purity. Versions with excess Al<sub>2</sub>O<sub>3</sub> (**PURALOX MG 20**) or MgO (**PURALOX MG 30**) are also available.

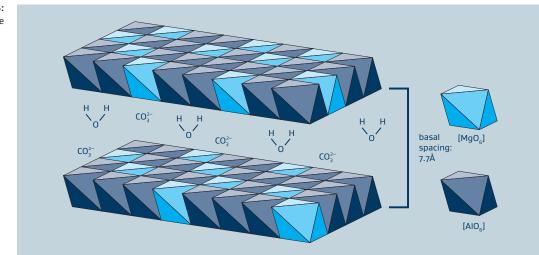


Figure 5: Crystal structure of hydrotalcite

#### 2.3. Doped aluminas

Our **PURALOX**, **SIRALOX** and **PURALOX MG** grades are also available as doped or even multi-doped versions with a large flexibility regarding the dopants, e.g. oxides of alkali, alkaline earth, rare-earth, various transition metals or selected phosphates.

Furthermore, even the homogeneity of the dopant distribution among the base support can be adjusted due to our flexible doping technologies. As an example, figure 7 shows an SEM-EDX intersection image of a lanthanum-doped alumina with a very homogeneous distribution of lanthanum among the alumina particles.

These materials can be tailored according to customer request. In case of interest, please contact a representative.

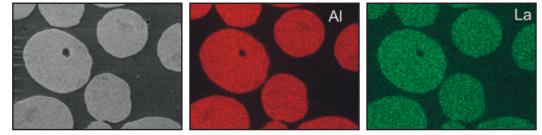


Figure 6: As an example figure 6 shows SEM-EDX illustration of a La-doped alumina

#### 2.4 Surface modified alumina

Apart from homogeneous element doping, Sasol also offers the possibility to deposit dopants on the surface of the pure or already doped alumina. This allows to adjust surface chemical properties like acidity or even to combine the beneficial physical properties of alumina with chemical properties of other oxides. The technologies used for the preparation also allow surface modification of shaped carriers like spheres and extrudates.

# 3. Technical data

#### 3.1 Silica-aluminas

| Typical chemical<br>and physical properties |        | SIRAL 1 | SIRAL 5 | SIRAL 10 | SIRAL 20 | SIRAL 30 | SIRAL 40 | SIRAL 40<br>HPV | SIRAL 70<br>HPV |
|---|--------|---------|---------|----------|----------|----------|----------|-----------------|-----------------|
| $AI_2O_3$ / $SiO_2$                         | [%]    | 99/1    | 95/5    | 90/10    | 80/20    | 70/30    | 60/40    | 60 / 40         | 30/70           |
| LOI   | [%]    | 25      | 25      | 25       | 25       | 20       | 20       | 20              | 20              |
| Loose bulk density                          | [g/l]  | 600-800 | 450-650 | 400-600  | 300-500  | 250-450  | 250-450  | 100-300         | 100-300         |
| Particle size (d <sub>50</sub> )            | [µm]   | 35      | 35      | 35       | 35       | 35       | 35       | 35              | 35              |
| Surface area (BET)*                         | [m²/g] | 280     | 350     | 370      | 420      | 380      | 480      | 500             | 360             |
| Pore volume*                                | [ml/g] | 0.5     | 0.7     | 0.75     | 0.8      | 0.9      | 1.0      | 1.15            | 1.3             |

#### 3.2 Doped aluminas

| Typical chemical<br>and physical properties |        | PURALOX<br>SCFa-160 Ce20 | PURALOX<br>SCFa-190 Zr20 |         |         | PURALOX<br>TH 100/150 L4 |
|---|--------|--------------------------|--------------------------|---------|---------|--------------------------|
| Al <sub>2</sub> O <sub>3</sub>              | [%]    | 80                       | 80                       | 97      | 96      | 96                       |
| Na <sub>2</sub> O                           | [%]    | 0.002                    | 0.002                    | 0.002   | 0.002   | 0.002                    |
| La <sub>2</sub> O <sub>3</sub>              | [%]    | -                        | -                        | 3       | 4       | 4                        |
| CeO <sub>2</sub>                            | [%]    | 20                       | -                        | -       | -       | -                        |
| ZrO <sub>2</sub>                            | [%]    | -                        | 20                       | -       | -       | -                        |
| L.O.I.                                      | [%]    | 3                        | 3                        | 2       | 2       | 2                        |
| Loose bulk density                          | [g/l]  | 600-800                  | 600-800                  | 500-700 | 550-750 | 300-500                  |
| Particle size (d <sub>50</sub> )            | [µm]   | 30                       | 30                       | 30      | 30      | 40                       |
| Surface area (BET)                          | [m²/g] | 160                      | 190                      | 140     | 145     | 150                      |
| Pore volume                                 | [ml/g] | 0.5                      | 0.5                      | 0.5     | 0.5     | 0.8-1.0                  |
| Pore radius                                 | [nm]   | 8                        | 5                        | 8       | 7       | 11                       |
| Thermal stability:                          |        |                          |                          |         |         |                          |
| Surface area: 24h/1100 °C                   | [m²/g] | -                        | -                        | 80      | 90      | 80                       |
| Surface area: 24h/1200 °C                   | [m²/g] | _                        | -                        | 40      | 40      | 50                       |

\*After activation at 550 °C for 3 hours.

Above Silica-Aluminas are also available as calcined materials under the trademark SIRALOX.

Further specialty grades are available upon request.

Analytical methods see page 14.

#### **3.3 PURAL MG/PURALOX MG**

| Typical chemical<br>and physical properties |        | PURAL<br>MG 5                                  | PURAL<br>MG 20           | PURAL<br>MG 30           | PURAL<br>MG 63 HT | PURAL<br>MG 70 | PURALOX<br>MG 26/100 |
|---|--------|--|--------------------------|--------------------------|-------------------|----------------|----------------------|
| Al <sub>2</sub> O <sub>3</sub> *            | [%]    | 95   | 80                       | 70                       | 37                | 30             | 74                   |
| MgO*  | [%]    | 5  | 20                       | 30                       | 63                | 70             | 26                   |
| CO3 <sup>2-</sup>                           | [%]    | -  | -                        | -                        | 10                | -              | -                    |
| LOI   | [%]    | 25   | 35                       | 35                       | 40                | 40             | max. 5               |
| Loose bulk density                          | [g/I]  | 300-600  | 300-600                  | 300-600                  | 100-300           | 350-500        | 250-400              |
| Particle size (d <sub>50</sub> )            | [µm]   | 40   | 40                       | 40                       | 3–9               | 40             | 30                   |
| Surface area (BET)*                         | [m²/g] | 250  | 250                      | 250                      | 15                | 180            | 100                  |
| Pore volume*                                | [ml/g] | 0.5  | 0.5                      | 0.5                      | n.a.              | 0.2            | 0.5                  |
| Speciality                                  |        | Basic Al <sub>2</sub> O <sub>3</sub> precursor | Al-rich spinel precursor | Mg-rich spinel precursor | Hydrotalcite      | Hydrotalcite   | Phase pure spinel    |

\*After activation at 550 °C for 3 hours.

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## 4. Product information

#### 4.1 Storage and transfer

Alumina powders are mildly abrasive materials having a Mohs hardness of 3.5 to 4.0. Therefore, handling and storage equipment should be designed for such materials. Examples are aluminium, abrasion resistant carbon steel and polypropyleneor epoxy-lined steel. Blower or vacuum systems can be used to move the powders. A minimum of 4,000 ft/min fluidizing velocity is recommended. Alumina powders will absorb atmospheric moisture, and facilities should be designed appropriately. The liquids are readily pumpable using standard centrifugal or positive displacement pumps. Due to the pH range of available products, recommended materials for process equipment include stainless steel, or polymeric-lined steel.

#### 4.2 Safety and handling

Pure alumina powders are classified as non-toxic nuisance dusts. Exposure to high concentrations of dust may cause physical irritation and drying of skin and eye tissues. Repeated or prolonged contact with alumina sols may cause irritation as well. Handling and shipping procedures should be designed to avoid such contact and to minimize the inhalation of airborne dust. Normal good laboratory practices and operating procedures should ensure personnel safety. For handling of different doped aluminas please see corresponding material data safety sheet.

#### 4.3 Technical support

The Sasol alumina organization is committed to offering the technical service necessary to ensure customer satisfaction. Technical support is available worldwide to aid you in choosing the best alumina for your needs, as well as for providing advice on safe and efficient use. The products described in this brochure give some indications of our total capability. We look forward to discussing specific technical requirements with you.







## 5. Certifications

All Sasol Chemicals locations worldwide are certified to DIN ISO 9001/14001 and to OHSAS 18001 standards (Occupational, Health and Safety Assessment Series), and the German plants additionally comply with EMAS III (Eco Management and Audit Scheme).

Our production sites operate according to an internationally recognised, integrated quality, environmental and safety management system that has been established at the sites for many years.

## 6. Analytical methods

#### 6.1 Element analysis

Alumina powder is quantitatively brought into solution by using acids and then analyzed by ICP, atomic emission. Additionally, X-ray fluorescence spectroscopy is used.

#### 6.2 Identification of crystalline phases

Powdered samples of the alumina are analyzed by using X-Ray Diffractometry (XRD) on either a Siemens D5000 or a Philips X'Pert diffractometer. The resulting powder patterns allow for identification of the crystalline compounds of the materials.

#### 6.3 Particle size distribution

The particle size distribution of alumina may be measured by various instruments, namely, Cilas Granulometer 1064 supplied by Quantachrome, Malvern Mastersizer or Luftstrahlsieb (air sieve) supplied by Alpine.

#### 6.4 Surface area analysis\*

The surface area of the alumina is measured by using an instrument supplied by Quantachrome (Nova series) or by Micromeritics (Gemini series). The method entails low temperature adsorption of nitrogen at the BET region of the adsorption isotherm.

#### 6.5 Pore volume and pore size distribution\*

The porosity is measured by nitrogen adsorption and desorption using Autosorb instruments supplied by Quantachrome.

#### 6.6 Differential scanning calorimetry (DSC)

Netzsch STA 449C Jupiter, Setaram 92 or Perkin Elmer instruments may be used with a selected heating rate to obtain the exothermic and endothermic transitions of alumina. Additional test methods are available for other physical properties upon request.

\*after thermal treatment at 550 °C for three hours



### At your service

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