Comparison of Methods for Measuring the Particle Size Distribution of Smokeless Tobacco Products

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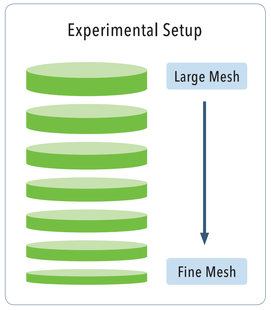
ABSTRACT

Tobacco cut size is one of the product properties for smokeless tobacco products which the Food and Drug Administration has proposed as a requirement for a Substantial Equivalence (SE) submission (21 CFR Parts 16 and 1107). However, no guidance has been provided for a standard method to perform these measurements. Sieve analysis has been used to determine particle size distribution (PSD) for smokeless tobacco products; however, this technique is time consuming, labor intensive, and requires that moist products be dried prior to sieving. Dynamic image analysis (DIA) is a method used to measure particle size that incorporates a high-speed camera to capture images of particles as they flow through a cuvette. Image analysis software is used to compute the PSD. DIA presents distinct advantages over sieve analysis. DIA has greater resolution, since the bin sizes for the distribution can be set much more narrowly than with sieves. DIA offers a wide particle size range, limited only by the camera and optics in use, which for this work ranged from 10 µm to 20 mm. The image analysis algorithms allow for a variety of metrics to be applied to the distribution. In this work, the particle size is calculated as the diameter of a circle of equal projection (EQPC), and length of fiber through a direct connection of the two most distant points (LEFI). The EQPC method allows us to easily transform the data from a length mode to surface area or volume weighted distributions. We examine differences in PSD of four smokeless tobacco products including snus, fine cut, mid cut, and long cut. A direct comparison between the weight-based sieve method and optical DIA methods is presented.

EXPERIMENTAL METHODS

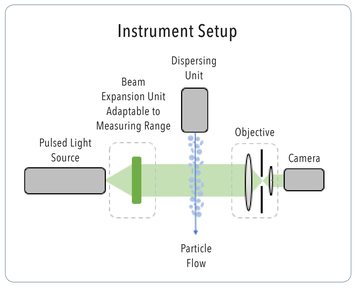
Sieve Method



- Based on draft CORESTA method (Smokeless Particle Size Analysis)
- Product passed through a U.S. Standard size 10 sieve to prevent clumping onto a pan and dried at 42 °C for 25 mins
- 50 g of dried snus (removed from pouch) or MST (Fine Cut, Mid Cut, and Long Cut)
- Uses U.S. Standard Sieves (sizes 18, 20, 30, 35, 40, and 45) with a catch pan and run on a Ro-Tap for 5 mins
- Each sieve was weighed empty as well as after sample processing on the Ro-Tap

Dynamic Image Analysis (DIA)

- Based on ISO13322-2:2006
- Sympatec QICPIC instrument paired with Lixell module pairing with peristaltic pump used
- Sample size in all cases was a heaping quarter teaspoon
- Sample soaked in 2.5 L DI water prior to running through instrument
- Results are calculated using desired method and images of all particles are stored in a library for any additional analysis



FACTORS USED TO CALCULATE PSD

Shape Descriptors

EQPC

- Based on the diameter (x) of a circle that is equivalent to that of a projection of the particle by the light source
- Uses the projection area (A) of a non-spherical particle:

$$x_{EQPC} = 2\sqrt{\frac{A}{\pi}}$$

LEFI

- Measures the length of a fiber from the two most distant points
- Uses "skeletonizing" to transform particle size to two dimensional line
- Good for fibers, but insufficient for spherical particles

Sphericity

- Sphericity is based on the ratio of the perimeter of the EQPC sphere (P_{EQPC}) to the perimeter of the particle (P_{real}) measured by DIA
- Given by the Equation:

$$S = \frac{P_{EQPC}}{P_{real}} = \frac{2\sqrt{\pi \cdot A}}{P_{real}}$$

- All calculations used a sphericity of $S = 0.9$
- Used to limit matrix effects (water) and eliminate counting air bubbles that form due to flow as particles

Calculated Sizes

- Density distributions, qr(x), are graphical representations of the particle size where the quantity of particles is displayed for each particle size fraction measured for analysis
- Cumulative distributions, Qr(x), graphically indicates the normalized fraction of particles which are smaller than the particle diameter, x, that is the portion referred to the total quantity
  - For r = 1, length is used
  - For r = 2, surface area is used (also called Sauter Mean Diameter)
  - For r = 3, volume is used

STRENGTHS AND LIMITATIONS

Sieve Method

- Particle sizes limited by standard sieve sizes (< 354 µm to 1 mm)
- Experiment is time-consuming and labor-intensive
- Fewer bins offers poorer resolution to calculate mean particle size
- Results are based on weight with limited options for data analysis
- Results can be biased based on the particle orientation as it passes through a sieve

Dynamic Image Analysis (DIA)

- Wide array of particle sizes limited by optics and camera (1 µm and 20 mm)
- Offers greater resolution due to narrow bin sizes
- Experiment is rapid with small sample size compared to traditional methods
- Image analysis algorithms allow for a variety of metrics to be applied
- Matrix effects due to flow are taken into account with sphericity

REFERENCES

1. ISO 13322-2:2006, "Particle Size Analysis – Image analysis methods – Part 2: Dynamic image analysis methods."
2. ISO 9276-1:1998(E), "Representation of results of particle size analysis – Part 1: Graphical Representation."
3. ISO 9276-1:1998(E), "Representation of results of particle size analysis – Part 2: Calculation of average particle sizes/diameters and moments from particle size distributions."
4. CORESTA Smokeless Tobacco Sub-Group Draft Method, "Smokeless Particle Size Analysis." (August 14, 2017)

RESULTS

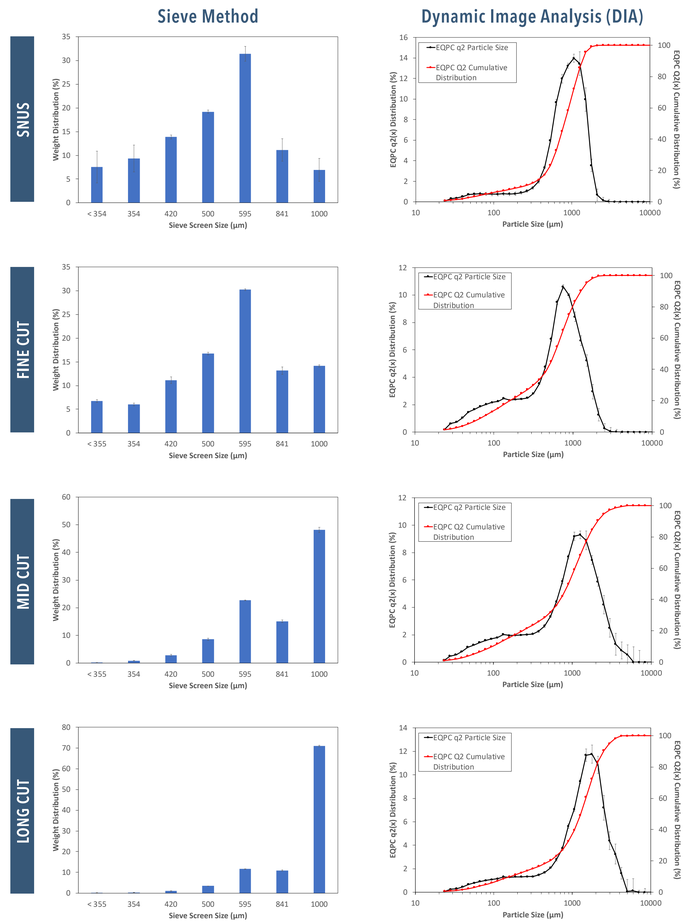


Table 1. Summary of mean values of particle size distributions for smokeless tobacco products

Product	Sieve Method (µm)	EQPC Q_2 (µm)	LEFI Q_1 (µm)
Snus	654 ± 52	819 ± 18	701 ± 136
Fine Cut	742 ± 8	627 ± 12	368 ± 12
Mid Cut	1105 ± 8	1021 ± 14	754 ± 12
Long Cut	1293 ± 2	1343 ± 33	1753 ± 73

CONCLUSIONS

- Welch's t-test (unequal variance) was run to compare sieve vs. LEFI Q1 and sieve vs. EQPC Q2 with $\alpha = 0.05$
  - -No statistical differences observed between both DIA methods vs. sieve method for snus and long cut as well as Q2 EQPC method for mid cut
  - -Statistical differences observed with both methods for fine cut and Q1 LEFI method for mid cut
  - -Difference can be accounted for higher variance of results using sieve method
- Each method has some bias based on how the particle size of smokeless tobacco products is calculated
  - -The sieve method is normalized by weight
  - -DIA used a high speed camera to capture images of particles that are later used to calculate the particle size through a variety of means based on projected area or length
- The advantages of DIA compared to the sieve method allow for a variety of metrics to be applied to the distribution while allow allowing rapid capture of data