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Influence of particle shape on size distribution measurements by 3D and 2D image analyses and laser diffraction

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ABSTRACT

This paper highlights the fact that particle size distribution (PSD) is not unique for the same product, and is dependent on the chosen measurement technique, especially for asymmetric shapes. Laser diffraction and 2D image analysis are commonly used PSD measurement techniques. However, the results may not be representative of the true physical dimensions of the particles.

The influence of particle shape on PSD results obtained from 2D/3D image analysis and laser diffraction was investigated. Two metallic powders presenting extreme shape properties (round and elongated particles) were analyzed, as well as a blend of the two pure products. 2D image analysis and laser diffraction results were compared to 3D image analysis (measuring the true particle size). This paper compares the PSD results obtained from the three methods.

Some commonly used size parameters in image analysis software did not give meaningful results in regard of the true physical dimensions of the particles. The existence of the two populations (products with extremely different shape and size characteristics) could not be identified with such size parameters, and laser diffraction also performed poorly. The PSD obtained from more precise size parameters (image analysis) better corresponded to the true dimensions of the particles.

This study highlights the strengths and weaknesses of particle size analysis techniques when studying products presenting diverse particle shapes, and points out that caution is required in the choice of the size parameters, and in the interpretation of PSD results.

1. Introduction

Sieving has been widely used for decades to calculate the particle size distribution of particulate matter. More recent techniques allow the investigation of new size ranges, and the measure of new size and shape parameters. Laser diffraction is a method routinely used in many industries, and image analysis instruments are also spreading rather quickly in the particle technology world.

The influence of particle shape on PSD results obtained from sieving has already been studied and the authors highlighted that the size distribution of a product was dependent on the shape of its particles [1–3]. Several authors also studied the relations existing between particle shape and particle size distribution obtained by laser diffraction [4–6].

However, for any particle size measurement technique, the obtained results are always a combination of the size and shape of the particles. No instrument can really measure the particle size distribution independently of particle shape. Although it is possible to obtain information about particle shape with laser diffraction [7], only image analysis allows the true characterization of particle size and shape. 2D image analysis gives only partial information on the particle size and shape, whereas 3D image analysis allows the measurement of the true characteristics of the particle.

In this paper, we discuss the ability of the selected measurement techniques (laser diffraction and 2D and 3D image analyses) to identify the existence of two populations in blends of differently shaped products. The influence of particle shape on particle size distribution results was also investigated. The true measured 3D dimensions of the particles allow the discussion of the accuracy of the other methods.

Blends of two products presenting extreme particle shape were prepared to address the matter. Two powders made of round particles and elongated particles were selected. However, the particle volume and density were comparable. Samples of the raw products were prepared, as well as intermediary blends of the two powders. X-ray microtomography was used for the acquisition of 3D images. 2D image analysis was performed with two different particle size and shape analyzers, and the particle size distributions of all samples were also measured by laser diffraction.

2. Materials and methods

The choice of the two powders was based on several criteria: nature of the material, volume of the particles, size of the particles and particle shape. Steel powders were selected, because the images

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obtained by 3D X-ray microtomography present a good contrast. Also, the density and volume of the particles are similar for both products. The types of the products were selected based on the manufacturers' specifications. For this research, we selected round steel particles (abrasive shot, trade name WS70, provided by Wheelabrator — France) and steel fibers (trade name AISI 434, provided by Stax — Germany). These two products present a great contrast in terms of particle shape, whereas the other features of the particles are similar.

Table 1 presents the physical properties of the two products.

Sections 2.2 and 2.3 present the 3D and 2D imaging techniques, as well as the measured size parameters. A nomenclature is presented in Section 5, as well as the correspondence with the ISO 9276-1 standard [14] 2D size parameters.

2.1. Sample preparation

Five different samples were prepared. Powder volumes of 200 cm^3 were prepared by mixing volume units of 50 cm^3 of the pure products. These volume units were prepared using a riffle sample divider and the powder volume was measured as an apparent, untapped volume of the raw product. Fig. 1 presents the volume fraction of round shot particles in each of the five samples, and the true calculated volume fraction of round particles and the round particle number fraction in each sample. These values were calculated from the data obtained by 3D image analysis. For each particle, a series of size and shape parameters were measured; a discriminant analysis algorithm [8] identifies if a particle belongs to the round shot category or to the fiber category.

The classification was performed with a representative set of 100 particles selected from the two pure samples. A test was executed on all the particles of the pure samples, and 99.9% of the particles were correctly classified. The classification allows to calculate the true volume fraction of round particles (the volume of each particle is measured), as well as their number fraction in the blends.

The observed differences come from the packing density of the two products, which is higher for round particles. Hence, for the same untapped volume of powder, the round shot sample contains a more important solid fraction than the fiber sample. Also, because the size range is wider for the fibers than for the round particles, the number fraction of fibers is more important in the blends.

For each sample in the series, subsamples were prepared for laser diffraction and 2D/3D image analysis. Subsampling was performed with a spinning riffler. The 200 cm^3 volume of powder is poured into 16 tubes on a rotating wheel. This process eliminates the segregation which may have occurred during the preparation of the five samples.

For laser diffraction and 2D image analysis, subsamples of 10 cm^3 were prepared. 3D X-ray microtomography requires only a very small amount of material (1 cm^3); hence another subsampling step was performed with a sample splitter.

A good contrast on the 3D images was obtained by mixing the metallic particles in a medium with a low atomic density. This is necessary to isolate images of each particle individually. This operation was carried out by mixing the metal powder with a polyethylene powder (<10% metal powder volume) in a cylindrical holder (PVC tube — 1 cm diameter). A sphere containing the tube was placed in a rotating tumbler for 30 min to disperse the metallic particles in the polyethylene powder. Two tubes were prepared for each subsample of the series.

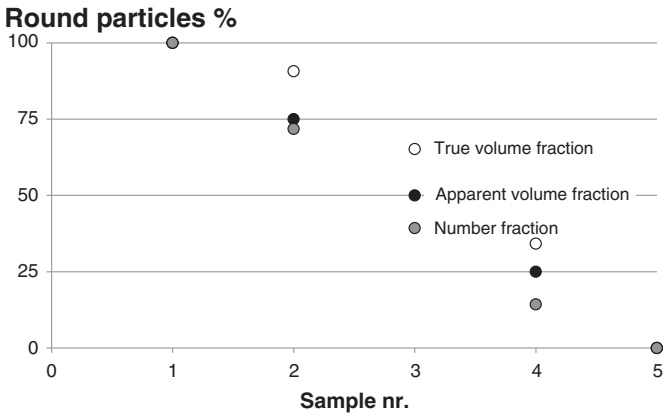

Fig. 1. The sample series consists of two pure samples of each product, and three intermediary blends. The prepared volume fraction (black dots) of round particles in the blends is different from the calculated true volume fraction (white dots) and from the calculated particle number fraction (gray dots).

The results presented in this paper are only those obtained for samples 1 (pure round shot sample), 3 (50/50 blend), and 5 (pure fibers sample), because they represent the most extreme cases.

2.2. 3D particle imaging

3D image acquisition was performed using desktop X-ray microtomography (Skyscan 1172). A sensor acquires projection images while the sample rotates; an algorithm [9] reconstructs slice images of the sample. 1000 slice images of 1200 × 1200 pixels were generated for each sample, with a resolution of 10 μm/pixel. The following operating conditions were applied: Source Voltage = 100 kV; Source Current = 100 μA; Image Pixel Size = 9.85 μm; and Filter = Al 0.5 mm. Because of the significant difference between the atomic densities of the steel particles and the polyethylene powder, an excellent contrast is visible on the images. The particles appear very bright and are easily isolated by a simple threshold algorithm that segments the gray-level 3D images.

This results in a binary 3D image (Fig. 2), processed with watershed and geodesy algorithms [10] to isolate touching particles and incomplete particles on the edge of the images. The final result is a 3D binary image with cubic voxels (3D image elements with a resolution of 9.85 μm in all three dimensions) containing the particles. Each particle is identified for individual size and shape analysis. For each of the five samples, between 1500 and 2500 particles were measured.

2.2.1. 3D size parameters

The first, most intuitional size measurement in the 3D space is the particle volume V (μm^3). This is the Lebesgue measure of a subset (particle) in 3D Euclidian space. In a discrete orthogonal grid, it is equal to the number of voxels of the particle multiplied by the volume of one voxel (9.85^3 μm^3). From the volume, the equivalent volume sphere diameter d_v can be calculated: $d_v = (6\,V/\pi)^{1/3}$. It is equal to the diameter of a sphere with the same volume as the particle. This parameter is a linear dimension, often used as a size descriptor. However, it does not represent a true physical size of a particle, in particular for particles with a shape departing from that of a sphere (Figs. 2 and 3).

Table 1
Physical properties of the particles (as provided by manufacturers). Fig. 3: 3D segmented cubes for samples 1, 2 and 3.

Product	Composition	Particle dimensions (μm)	Particle shape	Density ($kg.m^{-3}$)
WS70	Steel (Fe > 98.5%)	Diameter: 200–500	Round	7450
AISI 434	Stainless steel X6CrMo17-1	Diameter: 90–150 Length: approx. 1000 (max 3500)	I shaped, L shaped, U shaped…	7700