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Flowability characterization of nanopowders

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ABSTRACT

The applications of nanopowders are increasing significantly over the last years. In most of these applications, the flow behavior of the nanopowders seems to be a complicated, multiparametric but critical issue for the proper design of the processes. We have investigated, classified and compared several different metal oxide nanoparticles with respect to their flow properties. The flow properties of titania, silica and alumina hydrophilic nanopowders as well as their corresponding hydrophobic counterparts were determined by means of an annular shear cell powder flow tester (PFT). All the tested powders showed difficulties in flow while the titania nanopowders showed the highest difficulty among them. The results acquired regarding the compressibility, the flow functions and the effective angle of internal friction revealed that in all the cases the hydrophobic nanopowder seemed to be more cohesive than its hydrophilic counterpart. Moreover, the nanoparticles, no matter their polarity, showed negligible hygroscopicity while in the case of the alumina nanopowders the flow properties can be significantly influenced by ca. 1% (w/w) of moisture content.

1. Introduction

During the last decade nanoparticles have been attracting the interest of the scientific community more and more as the range of applications that they are involved in is increasing tremendously. The applicability of the nanoparticles is related to their particularities in properties which make them different from the properties of the conventional fine bulk solids. The properties of the nano-sized particles such us attractive forces, chemical, electromagnetic, rheological or optical properties are related to their size or to the high surface to volume ratio [1,2]. Moreover, nanoparticles often provide controlled functionality and increased reactivity which make them advantageous compared to other traditionally used materials in numerous industrial applications [3]. Nowadays, nanoparticles are widely used in solar cells, batteries, catalysts, pigments, cosmetics and other applications because of their unique properties [4].

Nanopowders are cohesive in nature, thus they naturally tend to easily form agglomerates with flow difficulties in terms of fluidizing [5]. In general, powders show a more complex behavior than fluids; nanopowders are even more intricate, and typically flow inefficiently in the industrial processes. Adhesive and cohesive forces between nanoparticles play a major role in their behavior. More specifically, effects of humidity, surface roughness, electrostatics as well as the molecular structure of adsorbate layers or the distribution of terminal groups on the particles' surfaces greatly influence the particle–particle adhesion behavior [6]. Liquid bridges between the particles are formed due to capillary forces that originated from adsorption and condensation of molecules on the particle surface, while the surface tension of the liquid and the geometry of the formed neck connector influence the cohesion force [7]. Moreover, depending on the polarity and the conductivity of the particles, the electrostatics, the van der Waals forces and the hydrogen bonding can be affected [7].

All the aforementioned phenomena regarding the interparticle forces are closely related to the flow behavior of the nanopowders. Knowledge of flow behavior in powders is a major concern in handling and processing operations such as flow from hoppers and silos, transportation, mixing compression and packaging [8]. Furthermore, flow properties are important in specific processes that nanopowders are involved in like fluidization and coating [2,9]. Unfortunately, although there are numerous synthesized nanopowders available and they are being used in many technological and industrial scale applications, there is a lack of quantitative data reported with respect to their flow properties. In 2014, Bouillard et al. carried out a rheological study regarding the cohesive carbon black and silica nanopowders with the use of a four-bladed vane powder rheometer [5]. Interesting results were obtained by their approach regarding flowability, cohesion energy, agglomerate sizes and dustiness. Recently, Kojima and Elliott published their study regarding the effect of silica nanoparticles on the bulk flow properties when mixed with fine cohesive powders [10]. Although their study was focused on the effect of various factors such as size, quantity and hydrophobicity of the silica nanoparticles on the flow properties of a model polymer powder, valuable information was acquired in terms of the flow properties of the individual nanopowders.

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The goal of this paper is to quantify, classify and compare several different metal oxide nanoparticles with respect to their flow properties. More specifically, the samples chosen for this study consisted of three different pairs of nanopowders based on different parental mineral moieties, titania, silica and alumina, while each pair was comprised of one hydrophilic and one hydrophobic individual nanopowders. The polarity of the particles depends on the surface chemistry of the corresponding molecules. Hydrophilic particles contain hydroxyl groups on their surface, while the hydrophobic particles are manufactured by substituting the hydroxyl groups of the polar particles with organic moieties via a hydrophobization process [7]. The hydrophilic titania-based Aeroxide P25 (Evonik) is used as a catalyst carrier and as an active component for photocatalytic reactions, while its hydrophobic counterpart Aeroxide T805 is used as an additive for toners and heat stabilizer for silicone rubbers. The hydrophilic silica-based Aerosil 130 is being used for providing reinforcement and thixotropic behavior in sealants, while Aerosil R972 is mainly used in applications such as silicon sealants by taking advantage of its hydrophobic properties. The hydrophilic alumina nanopowder Aeroxide AluC applications are related to its surface charge and optical properties, while Aeroxide AluC805 is used to regulate flow properties and protect the coated powders from moisture due to its hydrophobicity.

The flow behavior of the nanopowders was determined by means of an annular shear tester (powder flow tester — PFT) and the flow properties were evaluated by measuring the bulk densities, the flow functions and the effective angles of wall friction under different major principal consolidation stresses.

2. Materials & Methods

2.1. Powders

The working materials of the present study were nanopowders obtained from Evonik Industries AG. The details of the nanopowders, produced via flame synthesis, are listed in Table 1. Three hydrophilic–hydrophobic pairs of nanopowders were tested. The parental metals of the nanopowders were titanium, aluminum and silica.

The first step of our experimental procedure was the sieving of the powders in order to remove the relatively large agglomerates which can influence the bulk density and flow measurements. The sieving of the powders was carried out with the use of 335 μm pore diameter sieves placed on a shaker for 30 min before every measurement.

2.2. Determination of Flow Properties

The flow properties of powders of all the nanopowders were determined with the use of a powder flow tester (PFT) from Brookfield Engineering Laboratories Inc. PFT complies with the test procedure ASTM D6128 using the annular and Jenike's shear test techniques. The powder flow function is a plot of the unconfined failure strength versus the major principal consolidation stress.

A vane lid was used in the PFT measurements of the flow functions of the powders. The flow function tests were undertaken using the 263 cm^3 volume shear cell and running the standard flow function test program. The sample of each nanopowder was carefully loaded after the sieving procedure in the shear cell of the equipment. The powders were inserted in the trough with the use of a powder scoop aiming to the minimum compaction during sample preparation. The program measures the flow properties over the range of five major principal consolidation stresses in a geometric progression that generates values of circa 0.6, 1.2, 2.5, 5.0 and 10 kPa. PFT was connected to a PC provided with Powder Flow Pro V1.2 software.

A flat wall friction lid was used in the measurement of the bulk density of the powders. The bulk density tests were undertaken using the 263 cm^3 volume shear cell and running the standard bulk density test program. This program measures the densities over the range of five major principal consolidation stresses in a geometric progression that generates values of circa 0.02, 0.2, 0.5, 1.0, 2.0 & 5.0 kPa.

For the wall friction measurements a flat wall friction lid and a 263 cm^3 volume shear cell were used. The standard wall friction program measured the effective angle of wall friction over the range of five major principal consolidation stresses in an arithmetic progression that generates values of circa 0.5, 1.0, 1.5, 2.0, 2.5, 3.0, 3.5, 4.0, 4.5, and 5.0 kPa. Flow function, bulk densities and wall friction measurements represent the average values of three independent measurements.

2.3. Nanopowder Acclimation and Moisture Content Measurements

For the needs of the present study, all the nanopowders were measured after equilibrating them in two different conditions. For the first measurements the powders were placed for 24 h in ambient conditions. The relative humidity and the temperature were circa 50% and 20 °C respectively. Furthermore, measurements of the flow properties of dehydrated nanopowders were performed. Prior to these measurements, all the powders had been placed in a laboratory oven where they remained at 120 °C overnight.

The water content measurements of the nanopowders were carried out by means of a Perkin Elmer TGA7 Thermogravimetric analyzer. Thermogravimetric analysis (TGA) is a type of testing that is performed on samples to determine changes in weight in relation to change in temperature and involves heating of a sample in an inert atmosphere and measuring the weight. The initial temperature of the furnace during the measurements was 25 °C and reached the final temperature of 600 °C with a heating rate of 10 $°\mathrm{C \cdot min^{-1}}$. The samples were being heated in a nitrogen atmosphere. Moisture content was calculated from the difference in values between the samples conditioned in ambient conditions and the dried samples.

3. Results and Discussion

3.1. Densities of the Nanopowders

Fig. 1a, b, and c shows the bulk density of the nanopowders at different levels of consolidating stress. This graph shows that for all investigated nanopowders the hydrophilic type has a lower density than its hydrophobic counterpart. In Table 2 the differences ($\Delta\rho_b$) between the poured bulk density and the bulk density under ca. 5 kPa of consolidation stress for each nanopowder as well as the relative increase of

Table 1
Specifications of the nanopowders used in this study.

Commercial name	Code name	Chemical formula	Polarity	Average particle size	ρ_p[a] [kg m^{-3}]
Aeroxide TiO_2 P25	TiO_2-P	TiO_2	Polar	21 nm	4000
Aeroxide TiO_2 T805	TiO_2-A	TiO_2	Apolar	21 nm	4000
Aerosil 130	SiO_2-P	SiO_2	Polar	16 nm	2200
Aerosil R972	SiO_2-A	SiO_2	Apolar	16 nm	2200
Aeroxide AluC	Al_2O_3-P	Al_2O_3	Polar	13 nm	3600
Aeroxide AluC805	Al_2O_3-A	Al_2O_3	Apolar	13 nm	3600

[a] Particle density (ρ_p) values from [7].