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Wet grinding of $CaCO_3$ with a stirred media mill: Influence of obtained particle size distributions on pressure filtration properties

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ABSTRACT

Chemical and process industries utilize stirred media mills for efficient fine grinding of solids. Stirred media mills, also referred to as stirred ball or stirred bead mills, generally have a good ability to produce fine particles with a relatively narrow particle size distribution. Wet grinding with vertical stirred ball mills is typically carried out for slurries containing particles smaller than 200 μm, such as industrial minerals and pigments. Filtration is an important unit operation, which is performed in order to reduce the water content of the slurry after grinding and concentration, and prior to drying and transportation. Pressure filters are used to obtain the maximum dryness of the product. A number of properties of solids affect the filtration performance. The particle size and shape, the width of particle size distribution, and the net surface charge of the particles are among the factors having a significant influence on the filtration characteristics.

In this study, the dependency of the pressure filtration properties of wet ground calcium carbonate (GCC) slurries on the grinding result obtained by a stirred media mill is investigated. After grinding, subsequent filtration experiments were carried out with a laboratory-scale pressure filter. Average specific cake resistances, porosities of the filter cakes, and cake compressibility parameters were determined from the filtration data. The results showed clear correlations between the particle size data and the related filtration properties. Interesting observations were also made regarding the average porosity of the filter cakes and the uncertainty of the experiments.

1. Introduction

The separation of mineral particles from a liquid in the industry is always affected by the upstream process conditions applied during size reduction by the grinding circuit. One of the most important characteristics of ground solid material is the particle size distribution (PSD) of solids, which depends on the material [1], and may have substantial influence on separation performance in cake filtration. In order to make filtration as easy as possible, the particles should ideally be as large as possible, perfectly spherical and of the same size [2]. Because of the given requirements for the mineral product, such as narrow size distribution of fillers and pigments [3], fine grinding is necessary in many cases, albeit excessive reduction of particle size should be avoided.

Fine and ultrafine wet grinding of minerals, pigments, coatings, pharmaceuticals and other materials using stirred media mills is increasing [4–6]. The size of these mills varies, from < 1 dm^3 to several m^3, depending on the application [7]. Unlike in the case of tumbling ball mills, grinding in stirred media mills is not limited by the critical rotational speed because the mill wall is stationary and the grinding media are kept in rapid motion by an axial stirrer. This enables high-energy intensity, which is beneficial when the primary aim is to obtain fine particles. Stirred media mills have the ability to produce even extremely fine powders [8,9] with narrow PSD. For the purpose of very fine grinding, stirred media mills are also energy efficient compared with tumbling and vibrating ball mills [10].

The operational parameters of stirred media mills have a significant influence on the properties of the ground powder product. The most important properties of the product include particle size distribution [3], the shape of particles of different sizes [11], and surface properties and interactions that determine the stability of the slurry [9,11]. The grinding medium consists of balls or beads, typically made of steel, glass or ceramics. Spherical media have been shown to facilitate grinding compared to non-spherical media [13]. In addition to factors related to equipment design (dimensions and geometry, etc.), the most important factors affecting the grinding results include, for instance, the stirring speed, ball diameter and density, charge ratio, and slurry density [14–16], and interactions between the variables [17]. More generally, the grinding result obtained in a certain time is closely related to the specific energy input of the mill [18,19], provided that the suspension is electrostatically stable and no particle agglomeration occurs [12].

Cake filtration is an important unit operation in a large number of different applications in minerals processing and chemical industries. Filtration is typically performed after grinding and enrichment operations. The

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objective of filtration is to remove excess liquid from the slurry to improve the overall economy of the process, e.g. by minimizing the amount of water that is to be evaporated in the final drying stage. When there is a need to produce as dry a cake as possible, pressure filters rather than vacuum filters are likely to be applied. Depending on the product requirements, cake washing, squeezing and desaturation of the cake by the use of pressurized gas can also be performed in many pressure filters. The operational conditions in the filtration stage are selected on the basis of the properties of the slurry, i.e., a high-pressure difference is often helpful for relatively incompressible mineral cakes, while super-compressible materials are more readily filtered by using low pressures. Various particle sizes are used to characterize the PSD of the solids in the slurry to be filtered. For a monomodal distribution with a relatively regular shape, particle sizes of the undersize distribution, such as D_{10}, D_{25}, D_{50}, D_{75}, and D_{90}, are typically used to describe the distribution numerically. Additionally, the description of particle size can be done using the mean diameter of surface area distribution (D[3,2]), which is also called the Sauter mean diameter [20], and the mean determined from the volume-based distribution (D[4,3]) [21]. In theory, the Sauter mean diameter is also related to the filtration theory, for instance, by the Kozeny–Carman equation. The prediction of filtration properties on the basis of the properties of the slurry is difficult due to the numerous affecting factors and the interactions between them. Regarding the properties of solids, the most important parameters include PSD [22–25], shape [26,27], rigidity/deformability [28], and electrostatic stability [29,30], especially when small particles are present [2]. Recently, cake filtration has been studied to improve the understanding of the behavior of filter cakes during filtration and compression [31] and changes in cake porosity [32].

The aim of this study is to investigate the effect of fine grinding with a stirred media mill on the average specific cake resistances and other filtration properties of ground calcium carbonate (GCC) slurries in a pressure filter. GCC was ground under different conditions to produce slurries with various size distributions of the solids, to be able to associate the grinding results with the pressure filtration properties. The topic has not been widely discussed in the literature, in spite of its importance for the minerals and pigment industries. Laboratory-scale equipment was used in both the grinding and filtration stages to minimize the variance resulting from the experimental procedure.

2. Theory and calculations

At a glance, particle size distributions look relatively simple, but the complexity in the particle size measurement techniques and related calculations may cause misunderstandings when the results are finally interpreted. The particle characterization methods applied in this study are therefore described thoroughly. The filtration equations used for the calculation of the average specific cake resistance, cake porosity, and approximation of the cake compressibility are also introduced.

2.1. Particle characterization

Several different techniques have been developed for the measurement of particle size distributions. These techniques include, for instance, sieving, measurement of settling velocity of particles, laser diffraction, and direct image analysis. Because the number of small particles in mineral powders, including GCC, is much higher than the number of large particles, a number-based distribution cannot be used to describe the particle system properly. In this case, volumetric PSD is more applicable.

The diameters of D_{10}, D_{50}, and D_{90} practically mean that 10%, 50%, and 90% of the particles in the sample are smaller than these particle diameters. Considering a volumetric PSD instead of number distribution, D_{10}, D_{50}, and D_{90} represent particle diameters that divide the total volume of particles into similar volumetric classes as described above for the number distribution.

By converting the volumetric distribution to surface area distribution, the so-called surface mean diameter D[3,2], or Sauter mean diameter, is obtained. The definition of the Sauter mean diameter is given in Eq. (1) [33].

$$D[3,2] = \frac{\sum_{i=1}^{n} D_i^3 v_i}{\sum_{i=1}^{n} D_i^2 v_i} \quad (1)$$

where D is the diameter of a particle (m) and v_i is the proportion of particles in the size fraction. The Sauter mean diameter is inversely proportional to the volume-based specific surface area, which is defined by Eq. (3) below.

The volume mean diameter D[4,3] is calculated from the volumetric PSD according to Eq. (2) [33].

$$D[4,3] = \frac{\sum_{i=1}^{n} D_i^4 v_i}{\sum_{i=1}^{n} D_i^3 v_i} \quad (2)$$

The different particle sizes concerned in this study are illustrated graphically in Fig. 1. The example presented in Fig. 1 shows the measured representative particle sizes for the original GCC sample. As regards filtration, the presence of small particles in the slurry is typically the main cause for high cake resistances. It can therefore be assumed that the D_{10} and D[3,2] particle sizes may correlate best with the filtration characteristics. In the Results and discussion section of this paper, the different particle sizes presented in Fig. 1 are called representative particle sizes, to produce a difference between them and the characteristic particle size, which is sometimes used in filtration studies and can be solved from the Kozeny–Carman equation (see Eq. (8) below).

The specific surface area per unit volume S_v can be readily calculated for spherical particles using the Sauter mean diameter of particles [33]:

$$S_v = \frac{6}{D[3,2]} \quad (3)$$

The specific surface area relative to the mass of particles, referred to as S_w (m^2 kg^{-1}), can be calculated using Eq. (4) if the density of solids ρ_s (kg m^{-3}) is known.

$$S_w = \frac{S_v}{\rho_s} \quad (4)$$

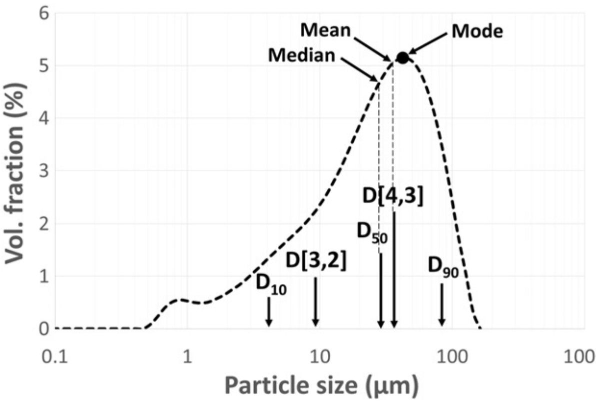

Fig. 1. Visualization of the different particle sizes used to describe the particle size distributions.