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Original Research Paper

Impact of grinding aids on dry grinding performance, bulk properties and surface energy

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ABSTRACT

In dry fine grinding processes the relevance of particle-particle interactions rises with increasing product fineness. These particle-particle interactions reduce the grinding efficiency and complicate the process control. The adsorption of grinding aid molecules on the product particle surface is a common measure to handle these effects. To ensure an efficient grinding aid application, the impacts of additives on particle and bulk properties, which influence the micro-processes inside the mill, need to be understood. Within this study the effects of several grinding aids on dry fine grinding of limestone in a laboratory vibration mill were investigated. Unlike in many other scientific works, the impacts of grinding aids were analyzed on different levels simultaneously: Grinding success and agglomerate size distributions were evaluated by wet and dry particle size measurements, respectively. Additionally, material coating on the grinding media, powder flowabilities and particle specific surface energies were measured. It was shown that all of the investigated grinding aids influence the grinding efficiency. However, the formation of agglomerates is not necessarily linked to the product fineness. Furthermore, a strong impact of certain grinding aids on the flowability of the product powder was determined. Thereby, the bulk flow behavior also determines the grinding result as it affects the stress mechanism inside the mill. Moreover, a direct relation between surface energy and powder flowability as well as agglomeration behavior could be demonstrated.

1. Introduction

In many industrial dry fine grinding processes, chemical liquid additives are added to the process in order to either increase the product throughput, decrease the specific power consumption or to reach a certain product fineness [1]. In some cases, these grinding aids are also used for improving the material-handling as well as for enhancing different product properties like cement strength and settling times [2,3]. Even though the benefits of grinding aids have already been shown in various experimental studies and industrial applications (e.g. [4]), there is still little knowledge about how the single components of commercial grinding aid blends affect the different product properties. Thus, the corresponding mechanisms of the interaction between particle surface and grinding aid molecules are not fully understood, which leads to empirically based grinding aid applications. The variety of applied substances including their different impacts (see e.g. [5]) complicate the development of a comprehensive understanding.

Nowadays, a direct impact of grinding aids on the fracture behavior of the particles is assumed to be irrelevant. The effects of grinding aids are rather attributed to adsorption phenomena on the particle surface, and thus, to a reduction of particle-particle interaction forces [6,7]. Several studies indicate that the benefits of grinding aids are mainly achieved by changing adhesive forces, the state of powder dispersion and powder flow characteristics [8,9]. In addition to that, Mishra et al. found a decrease of the agglomeration tendency of the product particles [7]. Since agglomeration can be assumed to be inversely proportional to the grinding performance [10], less energy is lost to the breakage of agglomerates and finer particles are produced at the same specific energy. Further, the grinding limit is shifted to a higher fineness and coating-effects on grinding media and mill equipment can be reduced [11,12]. Additionally, reduced adhesive forces result in a better dispersion of the product powder, improving the classification steps in grinding circuits since a smaller amount of fine particles is rejected as agglomerates and mistakenly led back to the mill [11,13]. Furthermore, by adding grinding aids a change in the angle of repose or several other flow characteristics was determined [11,14–16]. Moothedath and Ahluwalia showed that the changed

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flow behavior may affect the friction within the mill load while grinding. Consequently, the contribution of abrasion to the size reduction events is reduced at high grinding aid concentrations [14]. Independent of grinding aids, various authors [17,18] reported that the state of friction inside a planetary ball mill influences the ball motion, and thus, the stress conditions. Moreover, an improvement of material transportation inside the mill as well as a decrease of product retention times in continuous dry grinding processes were observed [11–13,19].

In the 1930s, Rehbinder [20] suggested that grinding aids also change the surface energy of the particles, making them easier to break. Even though this theory is nowadays assumed to be irrelevant for industrial grinding, the impact of grinding aids on surface energy is still being discussed (e.g. [10]). There are surprisingly little experimental findings about this theory available in literature. So far, only a few experimental attempts have been carried out to investigate the influence of surface modifications on the surface energy. For example, a decrease of both the dispersive component of the surface energy as well as polar surface properties was found in the case of calcium carbonate that was treated with stearic acid [21]. Similar impacts were also shown for a few other materials [22–24]. Arsalan et al. [25] showed that the total specific surface energy of dolomite und calcite is reduced when the particles are covered with water. Furthermore, Mishra et al. [7] determined a decrease of the agglomeration energy by molecular simulations after grinding aid applications. Those simulations showed that the adsorption of the grinding aid molecules happens with their polar parts. Thereby, hydrogen bonds as well as further polar interactions link the particle surface with the grinding aids functional groups. Consequently, an adsorption layer is formed keeping particles at a distance where the impact of short-range attractive particle-particle forces is reduced. Pasarín et al. [26] investigated the molecular ordering of adsorption layers of calcite surfaces in a gaseous atmosphere. In the case of linear molecules like ethanol, a highly ordered monolayer is formed directly on the solid surface. Outside of this layer, a further and more disordered layer with a thickness of two to three molecules builds up.

Apparently, grinding aids change various properties of the product powder, and thus, affecting the grinding process in different ways. In order to optimize a grinding aid application, the interactions of the grinding aid with the product particles and the resulting impacts on the product properties need to be known. Unfortunately, the variety of existing grinding aids, ground products, mill types and process parameters increase the challenge to achieve a comprehensive understanding. The aim of this work is to investigate the impacts of nine grinding aids from different substance classes on several product properties like particle size distribution, agglomerate size, specific surface area and powder flowability as well as grinding media coating. Additionally, the specific free surface energy of the product powder is analyzed using inverse gas chromatography.

2. Experimental

2.1. Vibration mill

An eccentric vibration mill of the type GSM 06 (Siebtechnik GmbH, Germany) was used for the grinding experiments. The mill is equipped with two identical, cylindrical 1 L grinding chambers made of steel. All operating parameters were kept constant in this examination. Each chamber was filled with 0.3 L of steel balls with a median diameter of 6 mm and a solid density of 7.86 kg/L. In order to obtain median particle sizes in the lower micron range (here: $x_{50,wet} < 10$ μm), the grinding time was set to 80 min in each experiment. After grinding, the total mill filling was sieved for ten seconds using a Retsch AS200 analytical screening machine and a 3 mm sieve. Then, the separated grinding balls were weighed in order to determine the amount of product particle adhesions, which was strongly bound on the grinding media.

2.2. Material

For the grinding experiments, calcitic limestone Durcal 40 (98% $CaCO_3$) by Omya, Switzerland with a median particle size of $x_{50} \approx 18$ μm was used. Solid and bulk densities of this material are $\rho_S = 2.73$ kg/L and $\rho_B = 1.074$ kg/L, respectively. Each chamber was filled with 198.1 g limestone which corresponds to a products filling ratio of $\phi_p = 1.5$. The limestone was dried for 20 h at 80 °C prior to the grinding in order to avoid an additional effect of moisture.

Within this study, nine chemicals (all Sigma Aldrich) from different substance classes were used as grinding aids (see Table 1). Several substances of these classes are established as grinding aids in commercially available grinding aid blends. Here, the particular components have been investigated in their pure form. The grinding aids were dosed into the grinding chamber prior to the grinding. Four different concentrations between 0.02 and 0.15 wt.% related to the mass of limestone were investigated.

3. Analysis

3.1. Particle size distribution

The particle size distributions of the product samples were measured by laser diffraction using a HELOS instrument (Sympatec, Germany). For the wet analysis, the powder samples were dispersed in ethanol and treated by ultrasonic waves prior to the analysis. Samples for the particle size analysis were taken from the total amount of ground material.

3.2. Blaine specific surface area

A manual Blaine device (Tonindustrie, Germany) was used to analyze the mass-related specific surface area of the product samples. In order to improve the statistical reliability of the results, three samples per test setting were analyzed in three-fold determination.

2.3. Agglomerate size

The agglomeration tendency of the product powders was evaluated using two different kinds of dry particle size measurements. First, the samples were analyzed with the HELOS instrument using the dry dispersion unit RODOS. Thereby, an air stream with a pressure of 0.4 bar was used to disperse the particles moderately.

A second type of agglomerate size was analyzed using an image analyzer QICPIC (Sympatec, Germany). The instrument was equipped with the dry dispersing unit GRADIS. This dispersing unit enables the measurement of dry samples with a minimum of agglomerate dispersion. Hence, mainly loose agglomerates are detected when analyzing powder samples with high product fineness.

3.4. Powder flowability

The powder flowability was analyzed using a ring shear tester RST-XS (Dietmar Schulze Schüttguttechnik, Germany) and a 30 ml shear cell. Measurements were performed at ambient conditions and repeated twice for every product powder. At the beginning of the shear test, the powder was pre-sheared under a normal stress of 10 kPa in order to load the bulk material with a

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