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Effect of operational parameters and stress energies on stirred media milling of talc

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ABSTRACT

Talc powder with a size of around 10 μm is used in many industrial applications such as in paints and coatings. A special feature of talc is its exceptional particle shape: platy with a three-layer structure. The properties for some applications are improved when talc platelets are dispersed to individual platelets in a wet stirred media mill. This study examined the effects of the operational parameters of a stirred media mill on the particle size and width of the particle size distribution (PSD) of talc. The aim was to find optimal operational parameters of a stirred media mill in order to minimize specific energy consumption and to reduce particle size. The particle size reduction of talc platelets was slow, due to the delamination step in the beginning of the grinding process. After delamination, the individual platelets were broken down, which was regarded as a stronger decrease in particle size as a function of specific energy. The PSD broadened as grinding proceeded, due to the platy shape of talc platelets. The smallest particle size with the lowest specific energy was obtained when the stress energy of the grinding media was around 100 μJ. The operational parameters at this point were the stirrer tip speed of 10 m/s and yttria-stabilized zirconium oxide beads with a size of 559 μm.

1. Introduction

Talc, which is composed of hydrated magnesium silicate or $Mg_3Si_4O_{10}(OH)_2$, is used in many industrial applications such as in paints, coatings, cosmetics, pharmaceuticals, papers, plastics, composites and as flame and ignition retardants [1–6]. A special feature of talc is its exceptional particle shape: platy with a three-layer structure in which octahedral magnesium oxide is layered between hydrophobic tetrahedral silica layers [1,6]. Talc particles have a high aspect ratio (particle diameter/thickness ≈ 20:1), referring to micron-sized dimensions of length and width and to the nano-sized dimension of thickness [7,8]. Due to this special property, talc qualifies as a good reinforcement and coverage filler of applications, compared to other industrial minerals such as calcium carbonate ($CaCO_3$).

The usage of small talc particles has been studied in different applications, including reinforcement of thermoplastic starch films [7], food packaging bags [9], binary and ternary nanocomposites [10,11], and semi-crystalline plastics [12]. The smaller size of talc particles has been found to provide better properties for all these applications. For instance, DePolo and Baird [13] studied the difference between fine talc and nanotalc in PC/PBT (polycarbonate/poly(butylene terephthalate)) composites and found many benefits of nanotalc, compared to fine talc. Additionally, Song et al. [14] ground talc particles with sand mill and observed an increased bending strength of the polypropylene matrix after grinding process. The use of talc in hybrid barrier films, together with nanofibrillar cellulose (NFC), was also examined in a few papers [15–17]. When the talc particles were mixed with NFC as a form of platelet aggregates, the mechanical properties of talc hybrids were lower, compared to those of mica, for instance [16]. Instead, when talc platelet aggregates were dispersed as to individual platelets in a wet stirred media mill [17], much higher tensile strength and Young's modulus values were obtained for NFC-talc films.

Currently, talc particles are mostly finely ground by dry mills, such as the air jet mill [18]. The challenge in industrial, ultrafine dry grinding is that the milling limit is currently in the region of 1 μm, due to the agglomeration of particles. However, for nanoparticle production, a wet mode of milling is preferred since it has been found to produce a smaller particle size than dry milling [19]. Wet stirred media mills are widely used in many industries, and the mills' operation is studied for different materials, including $CaCO_3$ [20], titanium dioxide (TiO_2) [21], and fused corundum [22]. Wet stirred media mills make it possible for particles to be ground down to their theoretical grinding limit [20,23,24]. Stirred media milling of talc has not been carefully studied although improved properties after milling have been obtained [11,13,14,17]. Therefore, this study investigated the effects of operational parameters on the particle size and the width of particle size distribution (PSD) of talc in fine grinding with a stirred media mill. The aim was to find optimal operational parameters of a stirred media mill in order to minimize specific energy consumption and to reduce particle size. Grinding experiments were performed by using a laboratory-scale stirred media mill in circuit mode.

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2. Materials and methods

2.1. Materials

The experimental material was talc (Finntalc M05N from Mondo Minerals, Finland), received as a fine dry powder. In brief, talc is produced from mined talc rocks, impurities are removed by flotation, and the final product size is achieved with air jet milling. The chemical composition of Finntalc M05N is the following: Magnesium oxide MgO (31%), Silicon dioxide SiO_2 (60%), Aluminium oxide Al_2O_3 (0.5%), and Iron oxide FeO (2.2%). Sodium polyacrylate (from Kemira Oyj, Finland) of 0.4 wt.% was used as a dispersant agent, and sodium carboxymethyl cellulose (Finnfix 5 from CPKelco, Finland) of 0.8 wt.% was used as a wetting agent. The chemical dosage of the grinding aids was the percentage of the dry chemical by weight, relative to the weight of dry talc in slurry (wt.%). The pH value of the suspensions was controlled by 5 M sodium hydroxide (NaOH) to be 11 during the entire grinding time in order to ensure maximum stability of the suspension [2]. Yttria-stabilized (Y_2O_3) zirconium oxide (ZrO_2) (YSZ) of different sizes, zirconium oxide (ZrO_2) and glass (SiO_2) beads were used as the grinding media (see Table 1).

2.2. Stirred media milling

At first, grinding aids (i.e., sodium carboxymethyl cellulose and sodium polyacrylate) were mixed with distilled water and the pH level was adjusted to 11 with NaOH. Then talc powder was mixed (500 s^{-1}) with water and the chemical mixture, leading to the final suspension solids concentration of 20 wt.%. A laboratory-scale stirred media mill with a disk stirrer (Hosokawa Alpine 90 AHM hydro mill) was used for the grinding experiments. The mill had an effective chamber volume of 1.12 l, and a 0.1- or 0.2-mm screen was used for separating the product from the grinding media. The circuit mode of stirred media milling was used (see Fig. 1). The flow rate of the slurry was kept constant (700 g/min) and controlled by a feeding pump. To avoid wear, the grinding chamber wall was coated with polyurethane (PU) and a PU stirrer was used. The grinding time for each suspension was 240 min. Three different stirrer tip speeds were tested: 6, 8, and 10 m/s. The experimental conditions are listed in Table 2. The power was recorded every half-minute during grinding. Specific energy (E_m) was calculated as the power input integrated over the grinding time (t_G) and divided by the mass of dry talc (m), as shown in Eq. 1:

$$E_m = \frac{\int (P_n - P_0) dt_G}{m} \quad (1)$$

where P_n is the mean active power and P_0 is the no-load power without grinding media and suspension.

2.3. Analysis

A gas pycnometer (Micromeritics AccuPyc II 1340) was used to measure the densities of the grinding beads. A Beckman Coulter LS 13 320 particle size analyzer was used to measure the exact, grinding bead sizes (Fraunhofer diffraction model) and PSDs of the talc samples (Mie optical model with refractive index of $1.57 + 0.1i$). Samples were diluted with distilled water and kept in an ultrasonic bath for 2 min before analysis. A span value was calculated from the data to indicate the width of the PSD, $(d_{90} - d_{10}) / d_{50}$. The field-emission scanning electron microscopy (FESEM, Zeiss Ultra Plus) was used to visualize the feeding material and ground samples. The FESEM samples were prepared by diluting the initial sample to 0.01 wt.%, and one droplet of each sample was dried in an oven (50 °C) overnight. The dried samples were sputter-coated with platinum. Low voltage (5 kV) and a working distance of 5 mm were used when imaging the samples.

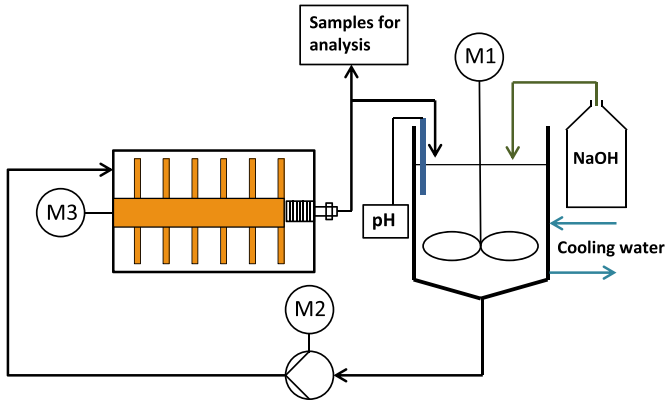

Fig. 1. Circuit mode of stirred media mill. The abbreviations in figure are the following: M1 is motor of stirrer in vessel, M2 is motor of feeding pump and M3 is motor of the mill.

3. Results

3.1. Feeding material

Fig. 2 illustrates the differential PSD of the feeding material (Finntalc M05N from Mondo Minerals). The particle size (volumetric median d_{50}) was 7 μm. The width of the PSD for the feeding material was 1.9.

3.2. Effect of grinding media size

The effects of different grinding bead sizes (d_{GM}) on particle sizes are presented as a function of specific energy in Fig. 3a. The grinding medium used was Yttria-stabilized zirconium oxide (YSZ), with bead diameters ranging from 240 to 1550 μm. The particle size reduction was slow in the beginning of the grinding process, and a greater decrease in particle size as a function of specific energy could be observed after grinding proceeded for 60 min (referring to 1000 kJ/kg). The smallest particle size with the lowest specific energy was achieved with a grinding medium size of 559 μm (Fig. 3a). Almost the same result was achieved with 870-μm grinding beads. The smallest and largest tested beads did not result in small particle sizes, compared to middle-sized beads, which achieved a higher specific energy and a smaller particle size. The effects of different grinding media sizes on the widths of the PSD are presented as a function of particle size in

Table 1
Grinding media sizes and densities.

Grinding media material	Grinding media density, ρ_{GM} [kg/m^3]	Grinding media size, d_{GM} [μm]
Yttria-stabilized (Y_2O_3) zirconium oxide (ZrO_2), (YSZ)	6100	239
		317
		559
		870
		1550
Zirconium oxide (ZrO_2)	3870	554
Glass (SiO_2)	2530	441

Table 2
Constant experimental conditions.

Item	Unit	Experimental condition
Filling ratio of grinding media, φ_{GM}	[vol%]	80
Solids concentration, c_m	[wt%]	20
Flow rate of suspension, $Q_{susp.}$	[g/min]	700
Dispersant dosage, D_{NaPa}	[wt%]	0.4
Wetting agent dosage, D_{CMC}	[wt%]	0.8
pH of suspension	[–]	11
Grinding time	[min]	240