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Original Research Paper

Effects of rotation speed and media density on particle size distribution and structure of ground calcium carbonate in a planetary ball mill

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ABSTRACT

Ultrafine ground calcium carbonate (GCC) produced by carbonate minerals is a widely used inorganic powder material. In order to get a finer GCC powder with narrow distribution span, effects of rotation speed and media density on particle size distribution and structure of GCC were studied. The grinding limit-particle size and distribution of GCC were measured by centrifugal sedimentation granulometer. The specific surface area, average particle size and structure of ground GCC were characterized by X-ray diffraction (XRD) and BET. The morphology of particles was observed by a scanning electron microscope (SEM). The result shows that low rotation speed and high-density media are conducive to obtaining a smaller particle with narrower size distribution. The crystal plane (012) and (122) of GCC are more stable than (018) and (202).

1. Introduction

Ground calcium carbonate (GCC) which is processed by mechanical methods using natural carbonate minerals such as calcite, marble, limestone etc. as raw material is an important inorganic non-metallic mineral. It is widely used in paper, plastics, paint, chemical fiber, rubber, coatings, pharmaceuticals, and fodder industries due to its superior properties in whiteness, thermal stability and rheological behavior. The quality of GCC like fineness, size distribution and purity has a great influence on its applications and product performance. For example, the product for paper coating must meet the requirements that the maximum particle size is not more than 5 μm, the content of particles smaller than 2 μm should be more than 95%, and the median size is between 0.3 and 0.5 μm [1]. And some paintings need the product whose D_{90} should be smaller than 3 μm, D_{50} is smaller than 1.3 μm, and specific surface area is bigger than 2.3 m^2/g [2]. Therefore, the study of GCC ultrafine grinding technology for effectively controlling the quality of products has significant importance.

It is well known that the superfine grinding process can be divided into two forms: dry and wet. Numerous previous literatures have studied the changes of size distribution, rheological behavior, surface properties, specific surface area and crystal structure of GCC ground in various wet equipment under different feed rate, rotation speed, solid mass concentration, media diameter and surfactant concentration [3–10]. Nevertheless, in practical production, one must separate solid and liquid phases firstly and then dry the particles after the wet grinding process. The separation is very difficult to perform and is accompanied by the loss of ultrafine powders. In addition, agglomeration frequently takes place after drying. Consequently, dry grinding is used more often than wet grinding [11]. The published studies on dry grinding of minerals are all concentrated on the impacts of milling time, bead size and rotation speed on GCC crystal size and structure in different dry mills [11,12]. As far as we know, no research has involved the effect of media density on the grinding process.

A planetary ball mill is used for ultrafine grinding and can be used in both dry and wet process. Because of greater energy density and higher grinding efficiency [13,14], comminution in a planetary mill is usually conducted at low rotation speed [15–20]. Ashrafizadeh and Ashrafizaadeh found that with an increase in rotation speed, the kinematic energy increases by a parabolic function and the variation of collision frequency of ball–ball are almost linear [16]. Fukumori et al., investigating the effect of rotation speed on size reduction, showed that as the rotation speed increased, the grinding rate became higher, but the particle size limit became larger [20].

Agglomeration is a prevalent phenomenon in superfine grinding [21–29]. In most grinding experiments, due to the agglomeration, a limit in particle size is reached where further grinding does apparently not lead to smaller particle sizes. Knieke et al. pointed out

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that the grinding limit can be divided into apparent and true, and the limit that is measured by the BET method is considered as true grinding limit [30]. Garcia et al. proved that the grinding media might cause product pollution during the process of superfine grinding, which would affect product application performance [5]. By ultrafine grinding calcite, Li et al. found that transformation of the crystal happens after a certain grinding time, and some peaks of aragonite appear in the XRD patterns [8].

In this paper, we investigate the grinding limit-particle size, size distribution, specific surface area, average particle size and phase transition of ground GCC under dry conditions in a planetary mill. The aim of this work is to study the effects of rotation speed and media density on particle size distribution and structure of GCC. The results might be of useful scientific value for practical production and utilization of ultrafine GCC.

2. Experimental

The initial GCC sample used in this work was provided by the Heng Liang New Material Technology Co., Ltd. The D_{50} and D_{97} of the sample were 13.51 μm and 58.58 μm respectively. A dry grinding test was carried out in a type CJXXM high-energy activation planetary mill (China–Russia High-Tech Incubator (Jiaxing) Co., Ltd). The planetary mill has two 130 ml pots, each of which has lining for the alumina ceramics. Every three minutes, an amount of powder was removed from the grinding pots and conserved to analyze, then grinding of the residual fraction was continued. The whole grinding time was set at 42 min. The operational parameters were as follows: the amount of GCC was 50 g; the weight of grinding media was 300 g; the rotation speed were 600 rpm, 800 rpm, 1000 rpm, 1200 rpm, respectively; the grinding media were zirconia (6.01 g/cm^3), alumina (3.62 g/cm^3), glass (2.45 g/cm^3) and zirconium silicate (4.41 g/cm^3) beads; the media diameter was 3 mm.

The particle size distribution was measured using a BT-1500 centrifugal sedimentation granulometer (Dandong Bettersize Instruments Ltd) in 0.2–150 μm. Every sample was measured three times, and the mean and confidence interval of these results was calculated. Before determination, GCC particles were treated with ultrasonic waves for 10 min.

The specific surface area of a sample was measured by JW–BK nitrogen adsorption surface tester (Beijing JWGB Sci. & Tech. Co., Ltd.). A representative amount of sample was taken from each sample, and then dried at 250 °C for one hour in order to remove the residual moisture of the sample before the measurement. Each sample was measured three times.

The mineral composition was analyzed using a D8 ADVANCE X-ray diffractometer (Bruker Co.). Each sample was scanned continuously at 4°/min from 10° to 60° at 35 kV and 20 mA in 0.01° steps. The qualitative identification of mineral phases was performed using the Jade 5 program. The profile fitting procedure was performed without smoothing the XRD spectra. The full-width at half maximum (FWHM), was obtained from the adjusted line profile. The errors were less than 3%.

The morphology of particles was measured using a field emission scanning electron microscope (S-4800, Hitachi, Ltd.).

3. Results and discussion

3.1. The influence of rotation speed on D_{50} and size distribution

In order to consider the influence of rotation speed, the media density was kept constant. The zirconia beads were used and the speed was increased from 600 rpm to 1200 rpm. The evolution of D_{50} is plotted as function of the grinding time in Fig. 1. It can be seen that the D_{50} curves have approximately the same trend, that

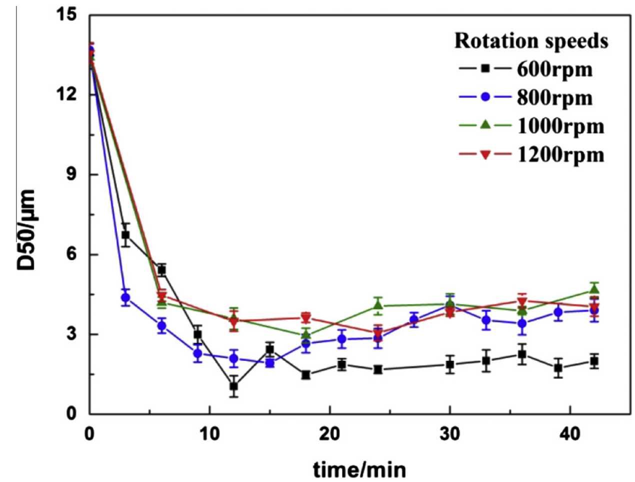

Fig. 1. The evolution of D_{50} versus grinding time (error bars: 95% confidence interval).

is, with increasing grinding time, the particle size decreases rapidly, then after a certain milling time no further decrease of the particle size occurs and a steady state is reached. Such a plateau in particle size was also found in other research [5,30]. This is probably due to the equilibrium between the fragmentation and the agglomeration processes. While as the rotation speed increases, the corresponding grinding limit-particle size becomes larger; a similar increase in limit-particle size has been reported by Fukumori from 440 rpm to 723 rpm [20]. When rotation speed varies from 600 rpm to 1200 rpm, the corresponding limit-particle size increased from 1.76 μm to about 4.5 μm. It can be concluded that increasing the rotation speed is not conducive to achieving a finer product. This may be due to the agglomerations formed primarily by fine particles. The agglomerations intensified with increasing rotation speed, and as a result the particle size as well as grinding limit increases.

Moreover, considering now the whole size distribution and not only the D_{50}, it can be observed in Fig. 2, as the speed increases the distribution curve moves to the right side, showing that increasing speed is not conducive to obtaining a refined product size distribution. Garcia pointed out that there are two kinds of agglomerates: some agglomerates made of fine particles and other agglomerates resulted from the coating of fine particles on coarser fragments [5].

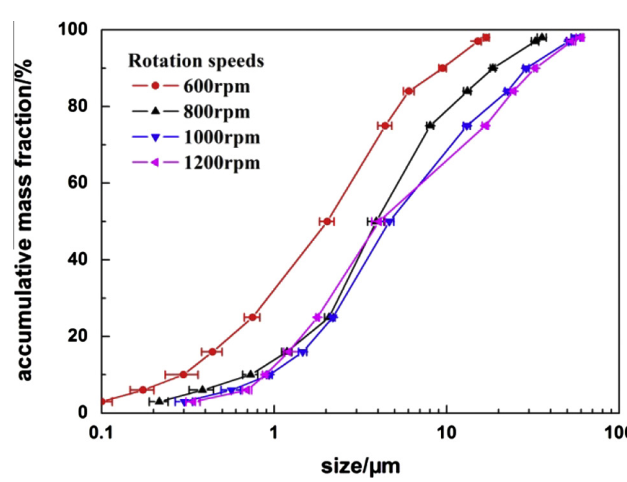

Fig. 2. Size distributions of ground GCC at grinding time of 42 min (error bars: 95% confidence interval).