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Effect of Operating Parameters on the Breakage Process of Calcite in a Stirred Media Mill

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Abstract

One of the most energy-intensive processes for producing submicron range calcite is stirred media mill. In the present work, numerous operating parameters such as solid mass fraction, grinding media size, media filling ratio, and grinding time have been investigated using a vertical type stirred media mill. The results are evaluated on the basis of mean particle size, specific surface area, and specific energy consumption. After conducting this study, optimum experimental conditions found to be as 70% media filling ratio, 25% solid mass fraction, 1 mm grinding media size, and 120 min grinding time. Besides, energy savings up to 22% were achieved with the choice of proper media size.

Keywords Stirred media mill · Calcite · Wet grinding · Submicron particles

1 Introduction

The demand for submicron particles and nanoparticles is increasing day by day. They have extensive applications in numerous fields such as paints, pigments, paper coatings, adhesives, catalysts, ceramics, water proofing materials, and printing inks due to small particle size and high specific surface area to volume ratios. There are generally two methods for production of submicron particles: (i) bottom-up in which the material is synthesized by means of chemical reactions, (ii) top-down in which coarse particle sizes are ground to produce fine particle sizes. What is more, wet grinding and dry grinding are two different plausible methods in the top-down approach. The stirred media mills have attracted intensive attention in recent years for grinding particles to submicron sizes because of several advantages such as low agglomeration trend, low material losses, less probability of oxidation, and elimination of the dust problem [1]. One of the most energy-intensive processes for submicron grinding is stirred media mills. Consequently, it is essential to optimize energy consumption by detecting the operational parameters for the stirred media mill [2]. Numerous investigations have been conducted on the production of submicron and nanoparticles of different materials in stirred media mill so far [3–7]. Nonetheless, little attention has been given to assessing the specific energy consumption depending on the operating parameters in the wet grinding process. It is known that a lot of parameters can affect the results of wet grinding and dispersion in stirred media mills. These parameters can be classified into four groups: grinding chamber and stirrer geometries, operating parameters (grinding time, stirrer speed, grinding media filling ratio, media size, properties, etc.), grinding operation mode (continuous, batch, pendular, or circulating mode), and suspension formulation (solid concentration, particle size, additives, etc.) [5]. The wet grinding technique has its own pros and cons regarding these parameters. Much of the published work reported on the effect of operating conditions in an attritor mill [3, 8–13].

Calcite powder ($CaCO_3$) is a salt, generally used in the submicron range, in paints, food, or pharmaceutical industries. Furthermore, it is used as mineral filler in the papermaking process since it makes the production of brighter paper possible with a greater resistance to yellowing and aging. Moreover, used as a part of the coating of the paper, it supplies larger opacity, printability, ink receptivity, and smoothness to the paper. It is also used as a filler in plastic industries to improve heat resistance hardness, color-fastness, or stability of the materials [14].

In this work, the possibility of producing submicron calcite particles with wet grinding in stirred media mill was

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Table 1 The chemical composition of the calcite used in experiments (wt.%)

$CaCO_3$	$MgCO_3$	SiO_2	Fe_2O_3	Al_2O_3	Total
99.5	0.2	0.01	0.01	0.02	99.74

investigated. Further, the effects of operating parameters (grinding media filling ratio, solid mass fraction, media size, and grinding time) on the production of calcite particles in stirred media mill were studied. The grinding results are evaluated by four parameters as the mean particle size (d_{50}), the increase of specific surface area (S_w), the reduction ratio (F_{50}/P_{50}), and the specific energy consumption (E_m).

2 Experimental

2.1 Material

Calcite ($CaCO_3$, $d_{50} = 5.4$ μm) powder obtained from Mikron's (Niğde, Turkey) was used for the experiments. Chemical and physical properties of the calcite samples are shown in Tables 1 and 2, respectively.

All the suspensions were prepared in the pure water. High-density (6000 kg/m^3) yttria stabilized zirconia (ZrO_2) grinding media (chemical composition 93% ZrO_2, 5% Y_2O_3, and 2% others) purchased from Cenotec Co., Ltd., Korea, were used for the submicron grinding experiments.

2.2 Stirred Media Milling

Grinding tests were carried out in a type of Standart-01 laboratory batch mill manufactured by Union Process (USA). The net volume of the grinding tank is 750 ml. To reduce the amount of wear caused by the materials of the mill, the grinding tank is made of ceramic (Al_2O_3). For the cooling purposes, the grinding tank is also equipped with a water jacket. The heat generated during wet grinding must be eliminated by circulating cooling water through the grinding container jacket. Both impact and shearing action cause size reduction and homogeneous particle dispersion with too little wear on the grinding tank.

The meaning of experimental conditions represented in Table 3 is described as follows. In grinding media filling ratio parameter column, for example, solid mass fraction, grinding media size, grinding time, and speed were kept constant, and the effects of different grinding media filling ratios (55, 60, 65, 70, and 75%) were investigated. After optimum grinding media filling ratio was determined, solid mass fractions (10, 15, 20, 25, and 30%) were investigated while keeping the remaining parameters at constant levels. As a result, the optimum solid mass fraction was determined. And the trials kept until all parameters' optimum levels are found.

These parameters were used in this study based on our former experiments and capacity of the mill [15].

2.3 Calculations

Equation 1 is used to calculate the grinding media filling ratio. This parameter makes a statement how much of the volume is filled with media using bed porosity of 0.4 [16], the value equals 100%.

$$J = \frac{\text{Mass of medias (gr)/Density(gr/cm}^3)}{\text{Mill volume (cm}^3)} \times \frac{1.0}{0.6} \quad (1)$$

The specific surface area is one of the basic properties of the sample and is generally represented by the total surface area of all particles contained in a unit mass of powder. The specific surface area (m^2/gr) (S_w) derived from Lecoq et al. [17] is:

$$S_w = 6/\left[\rho_s{}^* d(3,2)\right] \quad (2)$$

where ρ_s is the specific gravity for calcite (t/m^3), d(3,2) is the surface-volume diameter, calculated from the Wet Laser Diffraction Particle Sizer Malvern according to:

$$d(3,2) = \frac{\sum x_k dk^3}{\sum x_k dk^2} \quad (3)$$

where xk is the number fraction of detected size dk (number, %) and dk is the mean size of the detected class (μm).

The specific energy consumption during the wet grinding process was measured to evaluate the efficiency of the grinding process. The specific energy consumption (E_m) during the grinding process was calculated using the following equation:

$$E_m = \frac{E - E_0}{m_p} (\text{kWh/t}) \quad (4)$$

where m_p is the product mass, E is the energy used at the time t, and E_0 is the no-load energy.

Table 2 Physical properties of the calcite samples

Real density (kg/m^3)	Mohs hardness	Refractive index	d_{50} (μm)	d_{97} (μm)	Specific surface area (m^2/g)
2700	3.0	1.59	5.4	25.47	2.26

Table 3 Experimental conditions used in the wet grinding of calcite

Parameters	Grinding media filling ratio (J, %)	Solid mass fraction (%)	Grinding media size (mm)	Grinding time (min)	Speed (rpm)
Grinding media filling ratio (J, %)	55 60 65 70 75	20	1	120	600
Solid mass fraction (%)	70	10 15 20 25 30	1	120	600
Grinding media size (mm)	70	25	0.2 0.5 1 2 3	120	600
Grinding time (min)	70	25	1	60 90 120 180 300	600

The reduction ratio of calcite for each milling parameter was calculated from the following equation:

$$\text{Reduction ratio} = F_{50}/P_{50} \quad (5)$$

where F_{50} and P_{50} are the mean particle size of the feed (μm) and the product, respectively.

2.4 Analysis

A Laser Diffraction Particle Sizer Malvern 2000 Ver. 2.00 with Hydro 2000 MU attachment (Malvern Co., Ltd., UK) was used for the particle size analysis of the calcite feed and the ground products. The equipment can measure the particle size of suspensions and dry powders. Before determining the particle size, a representative amount of calcite samples was dispersed with ultrasound in 800 ml water for the measurements. Each test was repeated three times, and the values of measurements reported as the mean average. Malvern equipment can measure the particle sizes between 0.002 to 2000 μm under certain conditions. The energy consumed by the mill was measured by a voltomat-meter called Rev 2580 (Rev Ritter GmBH, Deutschland). In this study, only the active power per

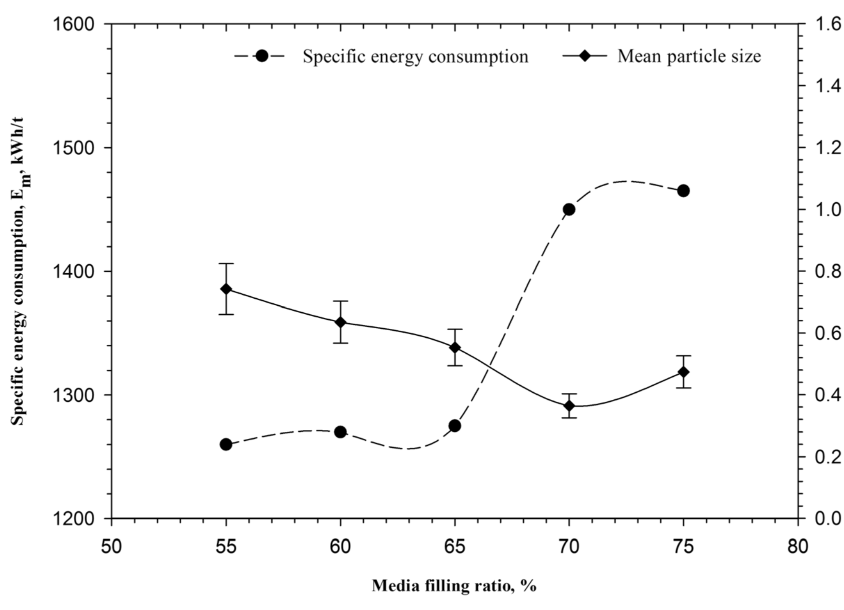
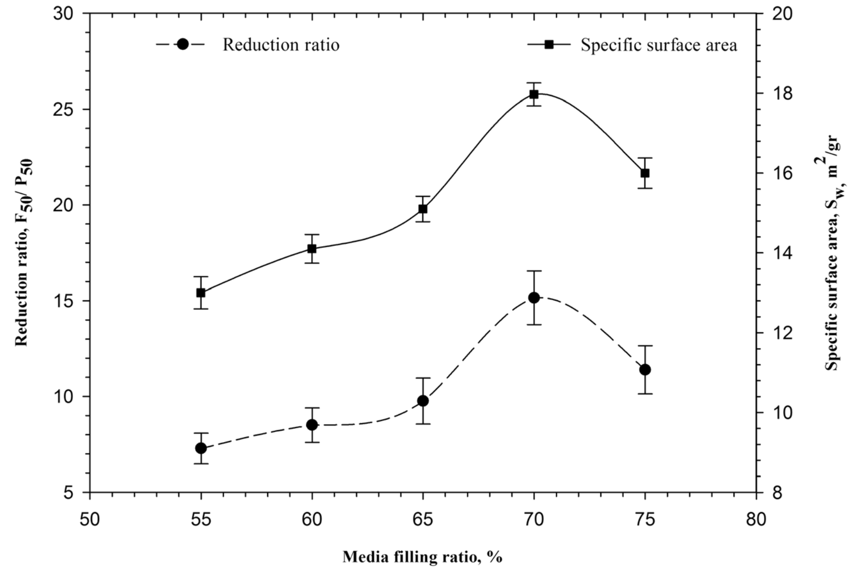


Fig. 1 Effect of media filling ratio on mean particle size and specific energy consumption

Fig. 2 Reduction ratio and specific surface area as a function of the media filling ratio



kilowatt hour was recorded. The SEM pictures of calcite feed and the ground products were obtained using a Zeiss Evo LS 10 microscope.

3 Results and Discussion

3.1 Effect of Media Filling Ratio

As mentioned before, the effects of five different filling media ratios (55, 60, 65, 70, and 75%) on the performance of calcite for wet grinding were investigated in this study. The effectiveness of five different media ratios can further be confirmed by observing media filling ratio effects on the produced mean particle size and specific energy consumption (Fig. 1). It is evident from Fig. 1 that a finer product size was obtained with increasing the media filling ratio. The increase in media filling ratio from 55 to 70% resulted in a rise in energy consumption from 1260 to 1450 kWh/ton and a reduction of mean particle size from 0.74 to 0.36 μm. Considerable difference in size reduction was not obtained with 55% filling where only a small difference was observed with 60% media filling. The best reduction was obtained with the 70% media filling ratio. After passing 70% media filling ratio, mean particle size has increased. By increasing the media filling ratio, particles move strongly and they are located along outer wall of grinding vessel. And then they show regular speed profile. Furthermore, the impact energy increases to a certain level of media filling ratio, and the particles' size decrease. While collision frequency between particles increases, collision energy decreases. Re-agglomeration may occur between fine particles at higher media filling ratio. As media filling ratio is further increased, possibly resulting in an increase of particle size [18]. Sadler et al. [19] changed the media filling ratio gradually and evaluated the loss in each size fraction for each case. They detected that better grinding performances were achieved when the mill was run at higher media filling ratios. The increase in the mean particle size attributed to re-agglomeration of the fine particles [20]. As media filling ratio increases, the number of media contacts increases, and the space between each grinding media decreases. In Fig. 1, the test results indicate that 55 and 60% media filling ratios are not energy-efficient conditions. In other words, higher energy consumptions are needed for lower media filling ratios to get the particle size reduction that is achieved by the higher media filling ratios (70%).

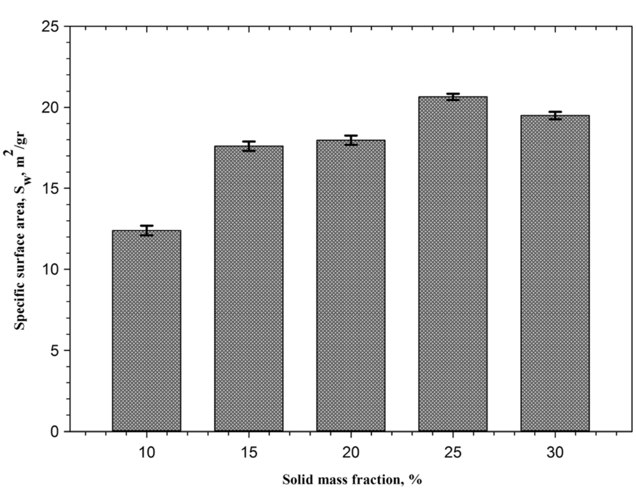


Fig. 3 Effect of solid mass fraction on specific surface area

The grinding behavior of minerals is often clarified in terms of the specific surface area of powders during

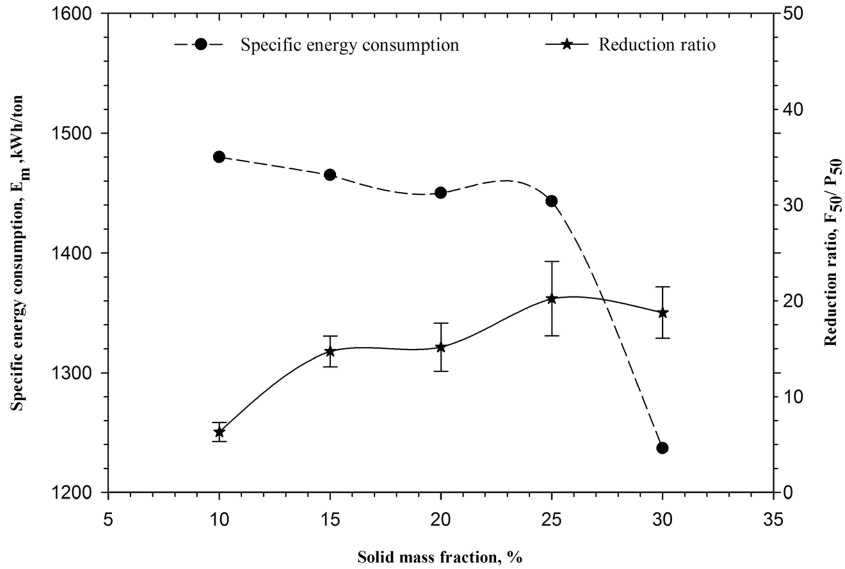


Fig. 4 Reduction ratio and specific energy consumption as a function of the solid mass fraction

grinding. Garcia et al. [21] indicated that the specific surface area increases a lot at the time very fine particles are produced when the mean particle size was determined on a volume basis. Reduction ratios and specific surface areas obtained for different media filling ratios are shown in Fig. 2. As can be seen, the increase in media filling from 55 to 70% has risen specific surface area from 13 to 17.97 m^2/g, but again the re-agglomeration phenomenon is monitored, and the specific surface area was decreased. Fuerstenau et al. [22] showed that the reduction ratio is dependent on the media filling ratio. It can be seen again in Fig. 2, with the high media charge, higher size reduction was achieved. However, after the 70% media filling ratio, a similar situation to that of reduction ratio and media filling ratio also occurred here as well.

3.2 Effect of Solid Mass Fraction

The solid mass fraction of dispersion in stirred media mills detects, how many particles are situated in a specific volume. In the event of stressing of a single particle, as the solid mass fraction increases, the number of stress for each particle and time decreases. Kwade and Schwedes [23] investigated the effects of the solid mass fraction and observed that increase in the solid mass fraction of the dispersions favors in the achievement of finer particle sizes. Increasing solid mass fraction affects the case of particles in two ways. On the one hand, the possibility that the particles are seized and pressured between the two grinding media is increased. This issue results in a better particle size reduction. Furthermore, the increase in the solid mass fraction of the suspension increases

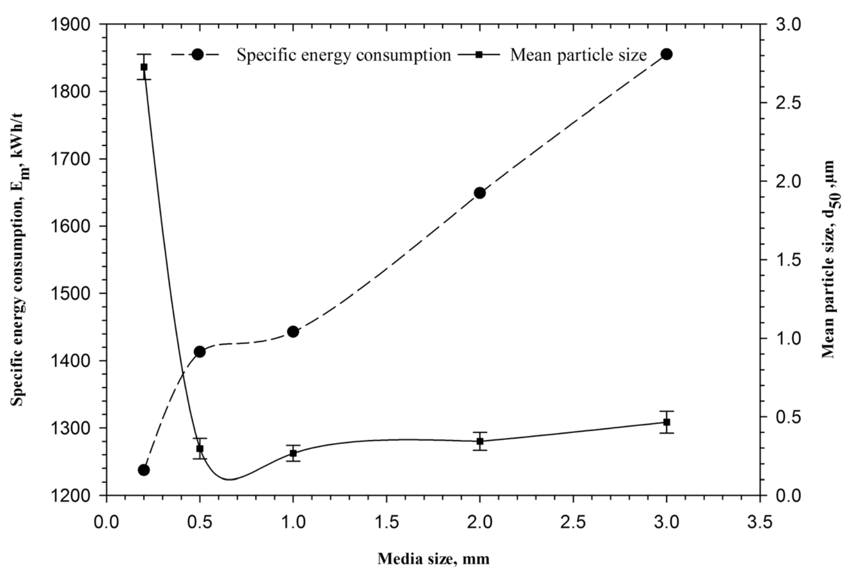


Fig. 5 Effect of media size on mean particle size and specific energy consumption

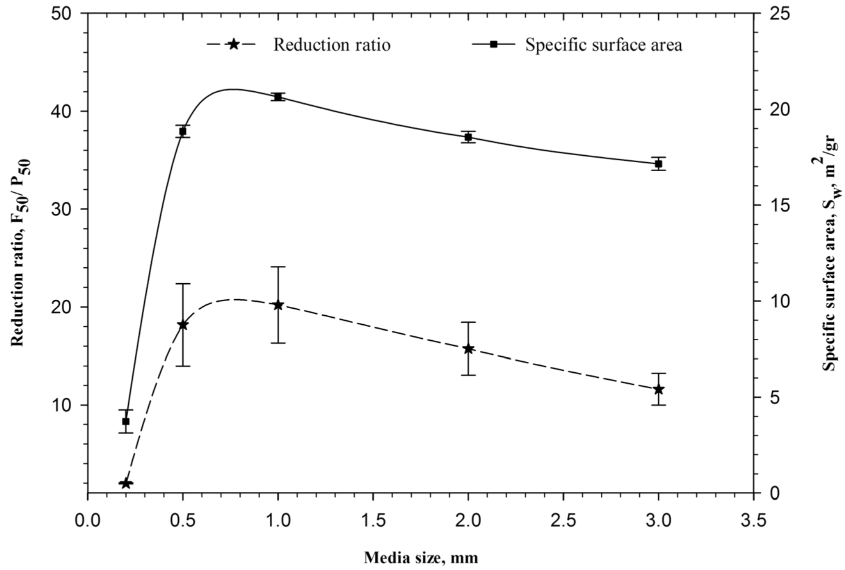


Fig. 6 Reduction ratio and specific surface area as a function of the media size

the viscosity of the suspension which may cause to agglomeration. In this study, several tests were carried out to investigate the effect of the solid mass fraction on wet grinding performance. The test conditions for investigating the effects of the solid mass fraction are given in Fig. 3. The figure shows the changes in the specific surface area as a function of the solid mass fraction. The surface area of the ground product is increased with the solid mass fraction. The increase in the solid mass fraction from 10 to 25% increased the surface area from 12 to 20.65 m^2/g. Nonetheless, after the 25% solid ratio, the specific surface area was decreased. This decrease was because the higher the solid mass fraction, the higher is the viscosity of the suspension. Zheng et al. [24] observed that the decrease of the specific surface area reveals that suspension (media and particles) around the center of impeller arms is stirred very well. However, beyond the impeller arms, the suspension remains nearly constant. Unground particles in that region cause a decrease in the product fineness and the specific surface area. Ding et al. [25] observed that for the low solid mass fractions, inter-particle distance is large enough to keep particles away from each other so that the particles can move in free motions, which, in turn, enhances suspension fluidity. A high solid mass fraction induces a short mean inter-particle distance, and the particle-particle interactions become more widespread. Therefore, the free motion of particles is disturbed [26].

Specific energy consumption decreases with increasing the solid mass fraction as shown in Fig. 4. The change in the solid mass fraction from 10 to 30% results in an energy consumption decrease from 1480 to 1237 kWh/ton. Kwade et al. [27] found that at low solids concentrations, when a particle is not exactly caught at each media contact, more specific energy is

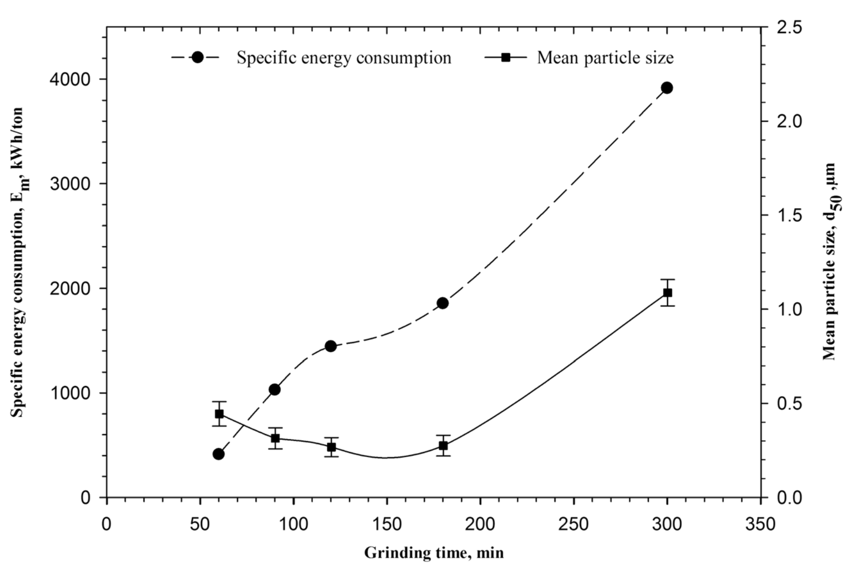


Fig. 7 Effect of grinding time on mean particle size and specific surface area

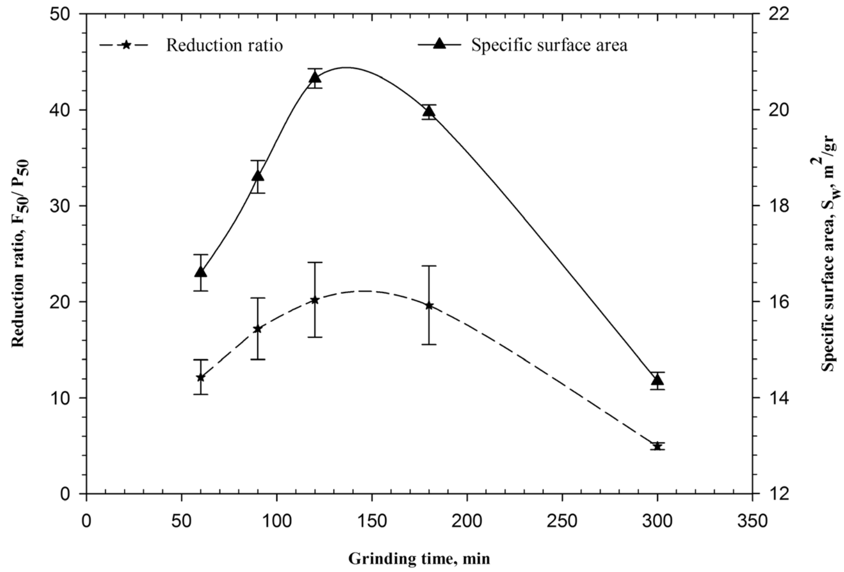


Fig. 8 Reduction ratio and specific energy consumption as a function of the grinding time

required to reach the same particle fineness. As the solid concentration increases, the fluidity of the suspension arises. For higher solids concentrations, difficulties arise in the fluidity of the suspension. From Fig. 4, it can be concluded that the best reduction ratio is achieved at the concentration of 25%.

3.3 Effects of Grinding Media Size

The choice of the proper size of grinding media is known to enhance the efficiency of grinding. As the media size decreases, specific surface area of the product becomes greater. This trend continues since the media size becomes too small to encompass particle fracture efficaciously. The use of finer media also ends up with reduced energy input because of the increased fluidity [28]. If the fine and coarse media are used in proper speeds, they are advantageous. Mankosa et al. [11] concluded that as the finer media was used, the effects of media size and the product size distribution became finer; hence, less energy was utilized. Furthermore, Kwade et al. [27], Wang and Forssberg [29], Jankovic [28], and Mende et al. [30] also monitored similar results in their studies. Bel Fadhel and Frances [31] observed that the use of finer media is limited to a certain extent. After this limit, separation of grinding media from suspension becomes a considerable problem. Altun et al. [32] also indicated that the use of the finer media is favorable over the coarser one, and 27% energy saving was accomplishable with this method.

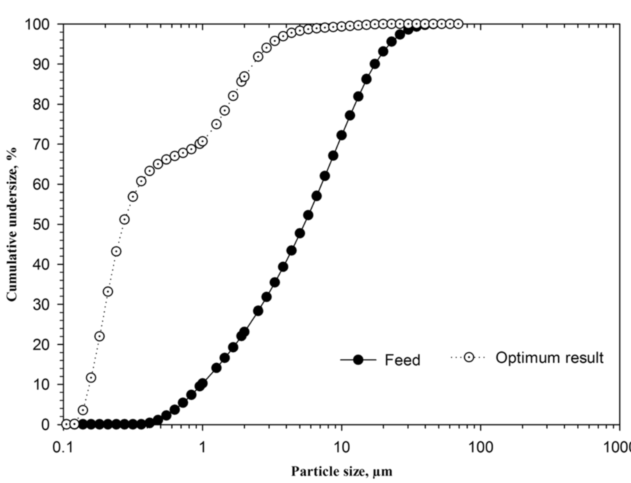


Fig. 9 Comparing the cumulative particle size distribution of the optimum result and the feed

In this research, the wet grinding performances of different media sizes as 3, 2, 1, 0.5, and 0.2 mm were compared, and the test results are indicated in Fig. 5. It is evident that the grinding media of size 1 mm is more efficient than 0.2 and 0.5 mm. Consequently, it can be said that 0.2 and 0.5 mm grinding media were not effective in this study. This can be explained that stirrer speed was affecting the performance of grinding. When finer media was used at a lower speed, the effect was so small. Finer grinding media was more efficient than coarser grinding media at high stirrer speeds. Jankovic [28] also determined that it is necessary to increase the stirrer speed to improve the grinding efficiency of smaller sized media. Nevertheless, 2 and 3 mm grinding media were also not as effective as 1 mm grinding media. The reason is, when coarser media was used at high speeds, the effect was very small. Coarser grinding media was more efficient than finer grinding media at low stirrer speeds. Finer particle size obtained with 1 mm grinding media was 0.26 µm in 120 min with an energy consumption of 1443 kWh/ton, whereas similar size was obtained with 2 and 3 mm grinding media with a longer time and higher energy consumption. This clearly shows that 1 mm grinding media produces finer particle sizes with less energy consumption as compared to 2 and 3 mm grinding media during wet grinding

Table 4 Comparing the optimum result and feed

	Feed	Ground
d_{10}(μm)	0.99	0.15
d_{50}(μm)	5.4	0.26
d_{97}(μm)	25.47	3.8
Specific surface area (m^2/gr)	2.26	20.65

for the same grinding time. The use of 1 mm grinding media at 600 rpm results in reduced energy consumption.

Figure 6 shows the changes in specific surface area and reduction ratio as a function of media size. The increase in media size from 0.2 to 1 mm increased surface area from 3.73 to 20.65 m^2/g. After the 1 mm media size, the specific surface area was decreased. In this parameter, finer mean particle size, high specific surface area, and high reduction ratio carried out with 1 mm media size.

3.4 Effects of Grinding Time

Grinding time has important effects on wet grinding. Toraman and Katırcıoglu's [15] investigation on the stirrer speed and grinding time showed that they have strong effects on the wet grinding efficiency. To study the effect of grinding time during the wet grinding process, experiments were performed at various grinding times (60, 90, 120, 180, and 300 min). The decrease in mean particle size and specific energy consumption with grinding time is shown in Fig. 7. The mean particle size gradually decreases with grinding time, yet after 120 min of grinding time, mean particle size was increased. Patel et al. [33] also observed that the mean particle size more and more decreases with an increase in grinding time. However, after 180 min of grinding, particle size appears to reach the highest point due to increased inter-particle interactions. After this point, it is not effectively utilized for breaking the particles of. It can be seen in Fig. 7 that as grinding time extended, specific energy consumption increased. At 120 min grinding time, specific energy consumption was 1443 kWh/ton.

In Fig. 8, the results of experiments carried out to compare the wet grinding performances of different grinding times are given. It can be seen that with the increase in grinding time, the specific surface area of the samples gradually increases but after 120 min of grinding, the specific surface area has decreased from 20.65 m^2/g to 14.3 m^2/g by the time. A similar situation also occurred between the reduction ratio and grinding time as well.

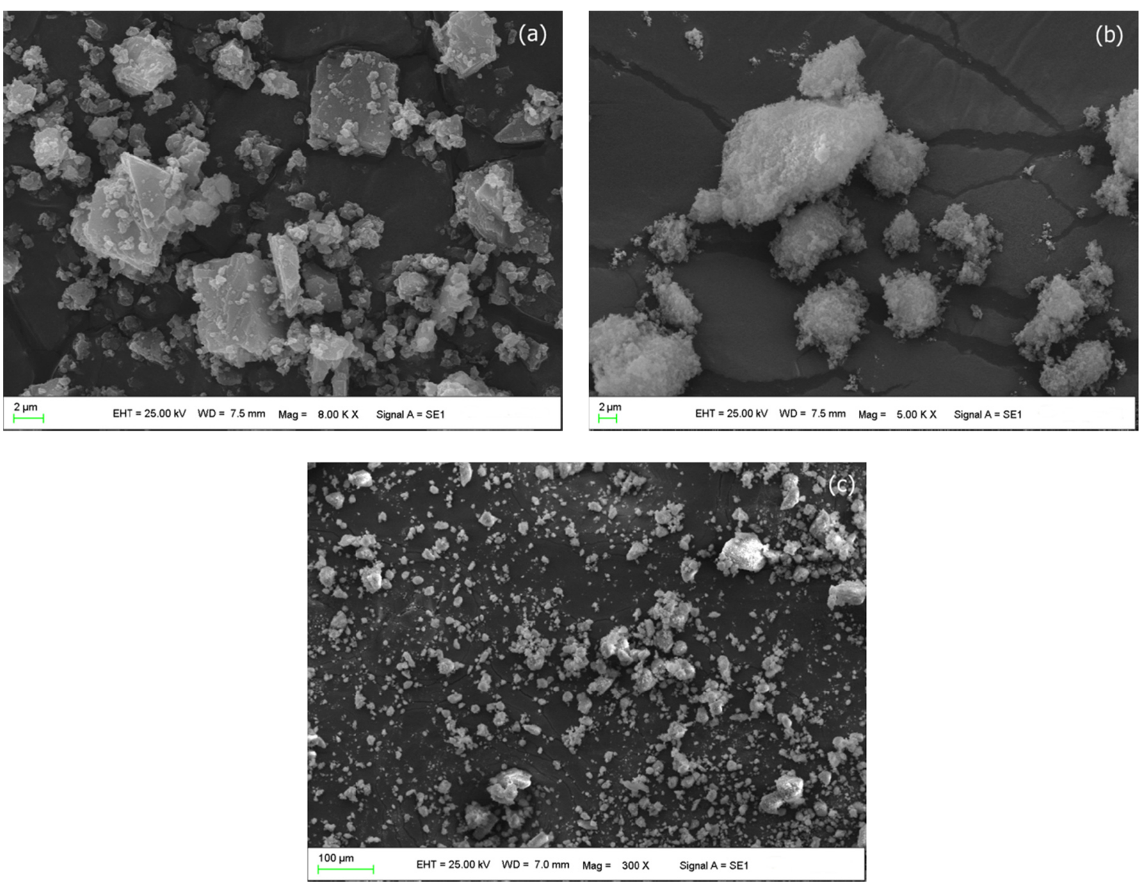


Fig. 10 Scanning electron micrographs of calcite: feed (**a**) and after 120 min grinding (**b**, **c**)

As a result of the grinding time tests performed at different grinding times, 120 min grinding was selected to be the optimum grinding time. Figure 9 and Table 4 show the relationship between the feed and the optimum result. The figure shows that the particle size distribution of the feed bimodal pattern and the optimum result is multimodal pattern [31]. Approximately 70% of the ground product is under 1 μm.

3.5 Effect of Grinding Process on the Particle Morphology

Calcite crystals are in the trigonal-rhombohedral structure. Typical images of this mineral are shown in Fig. 10. The irregular size distributions draw the attention before grinding, as illustrated in Fig. 10a. The scanning electron micrograph (SEM) image of Fig. 10b shows that agglomerates are made of fine particles on coarser particles. As long as the grinding progresses, their particle size decreases, while the proportion of the finer particle size increases as seen in Fig. 10c.

4 Conclusions

In this study, the effects of operating parameters such as media filling ratio, solid mass fraction, grinding media size, and grinding time were investigated. The results are discussed on the base of mean particle size, specific surface area, and specific energy consumption. The wet grinding tests carried out with stirred media mill showed that:

- Lower media filling ratio gave rise to inefficient grinding conditions. Namely, high energy consumption is needed to obtain higher size reduction values.
- Solid mass fraction affects the case of particles in two ways. First, the possibility that particles are captured and pressured between the two grinding medias, which results in better size reduction. Second, the increases in the solid mass fraction of the suspension, increase the viscosity of the suspension, which may cause to agglomeration.
- Choice of the proper size of grinding media enhances the efficiency of grinding. The selection of 1 mm media size was advantageous over 3 mm media size, and 22% energy saving was achievable.
- If the fine and coarse media are used in proper speeds, they are advantageous in terms of energy consumption and particle size distribution.

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Compliance with Ethical Standards

Conflict of Interest The authors declare that they have no conflict of interest.

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