

Optimization of Wet Grinding Parameters of Calcite Ore in Stirred Ball Mill

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Abstract

This study focused on ultra-fine grinding of calcite powder (CaCO_3) using a vertical stirred ball mill. The influences of various operating parameters such as stirrer speed (rpm), ball filling ratio (J), powder filling ratio (fc), solid ratio (wt.%) and grinding time were studied under wet conditions. The experiments were carried out in a batch mode of operation. The experimental results were evaluated based upon product size (d_{50}), specific surface area (m^2/g) and width of particle size distribution. As a result of this work, the optimum grinding test conditions were found to be 840 rpm for stirrer speed, 0.70 for ball filling ratio, 0.100 for powder filling ratio, 50% for solid ratio and 20 min for grinding time. It was also observed that stirrer speed, ball filling ratio and grinding time were directly proportional whereas powder filling ratio and solid ratio were inversely proportional to product fineness and specific surface area. The width of particle size distribution decreased with decreasing product size for all grinding conditions.

Keywords: Stirred mill, Wet grinding, Comminution, Ultra-fine grinding, Calcite, Shape of size distribution

Karıştırmalı Bilyalı Değirmende Kalsit Cevherinin Yaş Öğütme Parametrelerinin Optimizasyonu

Öz

Bu çalışma, kalsitin (CaCO_3) dikey karıştırmalı bilyalı değirmen de çok ince öğütülmesi üzerine odaklanmıştır. Karıştırma hızı (rpm), bilya doluluk oranı (J), malzeme doluluk oranı (fc), katı konsantrasyon oranı (%) ve öğütme süresi (dak.) gibi çeşitli çalışma parametrelerinin etkileri yaş öğütme koşullarında incelenmiştir. Deneyler, kesikli öğütme şartlarında gerçekleştirilmiştir. Deney sonuçları ürün boyutu (d_{50}), özgül yüzey alanı (m^2/g) ve tane boyut dağılımı genişliğine göre değerlendirilmiştir. Bu çalışma sonucunda, optimum öğütme test koşulları karıştırıcı hızı için 840 d/d, bilye doluluk oranı için 0,70, malzeme doluluk oranı için 0,100, katı oranı için %50 ve öğütme süresi için 20 dakika olarak bulunmuştur. Karıştırma hızının, bilya doluluk oranının ve öğütme süresinin ürün inceliği ve spesifik yüzey alanı ile doğru orantılı olduğu, malzeme doluluk oranı ve katı konsantrasyon oranı ile ters orantılı olduğu gözlenmiştir. Ayrıca, tüm öğütme koşulları için tane boyut dağılımı genişliği azalan ürün tane boyutu ile azalmıştır.

Anahtar Kelimeler: Karıştırmalı değirmen, Çok ince öğütme, Kalsit, Boyut dağılım şekli

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1. INTRODUCTION

Grinding, particularly ultra-fine grinding, is a unit operation with high energy requirements in the general process in which finer products with larger surface area are produced. Requirement of high energy input and inefficiency in grinding technology for materials such as chemicals, mineral, pigment, ceramic, cement, and food have long been considered to be area that needs improvement, especially for obtaining particles in the submicron size range [1]. In order to do this, varying types of grinding machines like ball mills, vertical roller mills, stirred mills and jet mills are employed in operations of regrinding, fine grinding and ultra-fine grinding.

Ultrafine particles can be produced on an industrial scale by either wet or dry grinding. The selection between dry and wet grinding depends on several factors. If the final material is used as dry powder, the slurry must be dried after milling. However, if the end product will be used as wet slurry or if the feed is already in the liquid phase, wet grinding is always a more economical process. While typical products of dry milling are in the range of 3-10 μm , wet grinding processes can exclusively produce higher fineness even in the submicron range. Such wet milled particles are utilized in paper processing as filler or coating pigment directly in the slurry [2]. Moreover, the chemical and pharmaceutical industries, in addition to the ceramic, paint or microelectronic industry, need more and more suspensions of materials with greater levels of fineness and storage stability. A way of obtaining these is wet comminution using the stirred ball mills [3]. Stirred ball mills have been increasingly used in the aforementioned industries for grinding particles down to the submicron range due to their operational characteristics such as easier operation, simpler construction, higher reduction rate, effective particle breakage mechanism, production of narrower size distribution and lower specific energy consumption in comparison to other fine grinding machines [4-9].

The purpose of this experimental study is to investigate the influences of operating parameters such as stirrer speed (rpm), ball filling ratio (J), powder filling ratio (fc), solid ratio (wt.%) and grinding time on wet milling of calcite (CaCO_3) by using a batch-operated laboratory type stirred ball mill. The experimental results were evaluated based upon product size (d_{50}), specific surface area (m^2/g) and width of particle size distribution.

2. MATERIAL AND METHOD

2.1. Material

The sample in this work was calcite powder (CaCO_3) (d_{50} : 41.49 μm , specific surface area: 1.03 m^2/g) from Nigde, Turkey with a density of 2.71 g/cm^3 . The chemical properties of the sample (by X-ray fluorescence) are given in Table 1. The size distribution of the feed sample is illustrated in Figure 1.

Table 1. Chemical composition of the sample (wt. %)

Species	%
CaCO_3	98.34
MgCO_3	0.97
Fe_2O_3	0.001
SiO_2	0.05
Al_2O_3	0.04
TiO_2	0.02
P_2O_5	0.02
MnO	0.006
Na_2O	0.11
SO_3	0.07

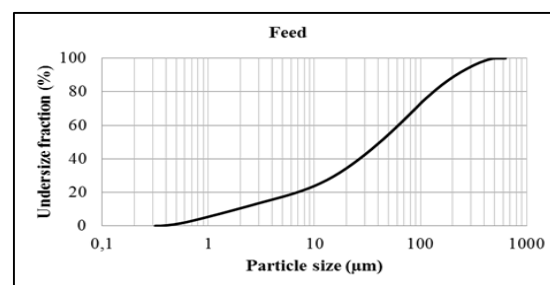


Figure 1. Feed size distributions of the calcite sample

2.2. Method

2.2.1. Vertical Stirred Ball Mill

The wet grinding tests were carried out in a vertical stirred ball mill with polyethylene liner inside which had a diameter of 150 mm and a height of 170 mm (Figure 2). The stirrer axis is fitted with four slotted shaft and arms (stainless-steel). The shaft is rotated with a motor which has a power of 0.37 KW, run at RPMs from 100 to 1400 and can be utilized for wet or dry milling. Alumina balls with diameters of 2.5-3.5 mm and a density of 3.6 g/cm³ were utilized as the grinding media.



Figure 2. The stirred ball mill

2.2.2. The Milling Conditions

The influences of operating parameters such as stirrer speed (rpm), ball filling ratio (J), powder filling ratio (fc), solid ratio (wt.%) and grinding time were investigated. In order to calculate the powder filling ratio (fc) and the ball filling ratio (J), Eqs. 1 and 2 were used. The wet grinding test conditions are outlined in Table 2. The grinding experiments were performed as a batch operation where the specimens were collected from the vessel at determined intervals of grinding time.

Following each of the experiments, all of the grinding media and the ground samples were taken out of the tank, and the grinding media and the products were separated by sieving.

Table 2. Wet grinding test conditions

Influence of parameter	Speed (rpm)	Ball filling ratio (J)	Powder filling ratio (fc)	Solid ratio (wt.%)	Grinding time (min)
Speed (rpm)	560	0.60	0.100	50	5
	840				
	1120				
	1400				
Ball filling ratio (°J)	840	0.40	0.100	50	5
		0.50			
		0.60			
		0.70			
Powder filling ratio (fc)	840	0.70	0.050	50	5
			0.075		
			0.100		
			0.125		
Solid ratio (wt.%)	840	0.70	0.100	35	5
				50	
				65	
				80	
Grinding time (min)	840	0.70	0.100	50	2
					5
					10
					15
					20

$$fc = \frac{\text{mass of powder} / \text{powder density}}{\text{mill volume}} \times \frac{1.0}{0.6} \quad (1)$$

$$J = \frac{\text{mass of balls} / \text{ball density}}{\text{mill volume}} \times \frac{1.0}{0.6} \quad (2)$$

2.2.3. Determination of Particle Size (d₅₀) and Specific Surface Area (m²/g)

Particle size (d₅₀: 50% cumulative weight passing size) and specific surface area (m²/g) are most commonly used variables describing the characteristics of ground calcite in many different industries such as dye, ceramic, paper and plastic, and they were utilized to evaluate the fineness of the calcite that was ground in this work.

The particle sizes and surface areas of the feed and the ground samples were analyzed using a Wet Laser Particle Sizer Malvern 2000 Ver. 2.00 with a Hydro 2000 MU attachment manufactured by

Malvern Co. Ltd., UK. The device has a measurement range of 0.020 to 2000 μm for particle size. A representative quantity of the samples was spread by ultrasonication in 800 ml water for the measurements. Three replications were carried out for each test, and the average values are reported.

2.2.4. The Width of Particle Size Distribution

A narrow particle size distribution is often desirable in the powder processing industry and at the mineral separation stage [10,11]. There are several evaluation methods describing the width of particle size distribution. Johnson et al. [12] described the width of particle size distribution using the ratio of d_{84}/d_{16} . Karbestein et al. [13], Jankovic and Sinclair [11] and Adi et al. [14] used the ratio of $(d_{90}-d_{10})/d_{50}$, Wang and Forssberg [10] used the Rosin-Ramler distribution, then the exponent of the equation, representing the slope, Nakach et al. [15] used the ratio of $(d_{90}-d_{10})/2d_{50}$, Nettet et al. [16] and Jankovic and Sinclair [11] used the ratio of d_{80}/d_{20} , Werner [17] used the ratio of d_{50}/d_{20} , and Kotake et al. [18] used the ratio of d_{90}/d_{10} . A decrease in these ratios means a narrower particle (product) size distribution. In this work, the shape (width) of the particle size distribution that was produced for each operating parameter was evaluated based upon the ratio of d_{90}/d_{10} . Additionally, the ratios of d_{80}/d_{20} , $(d_{90}-d_{10})/d_{50}$ and d_{50}/d_{20} were also discussed for all conditions.

3. RESULTS AND DISCUSSION

3.1. The Influences of Stirrer Speed

Higher stirrer velocity has an improved effect on grinding efficiency since the particle size is reduced. By increasing the speed of stirring, the velocities of the particles increase, which in turn led to more frequent and stronger clashes among the particles [19]. According to Kwade [20], the stress energy and the number of stress events depend strongly on stirrer velocity and grinding media size. The influences of four different stirrer speeds on d_{50} size and specific surface area are

presented in Figure 3. As the stirrer speed is increased, both the specific surface area and product fineness increased. It was observed by many studies that increasing stirrer speed produces a finer product and larger surface area [4,6,21].

It is also seen in Figure 3 that size reduction became insignificant at higher stirrer speeds (1120, 1400 rpm) due to the inefficiency of the grinding operation. This result agrees with those of Fadhel and Frances [22] and Altun et al. [23] who reported that the excess energy that is transformed into heat at higher stirrer speeds stops the grinding process.

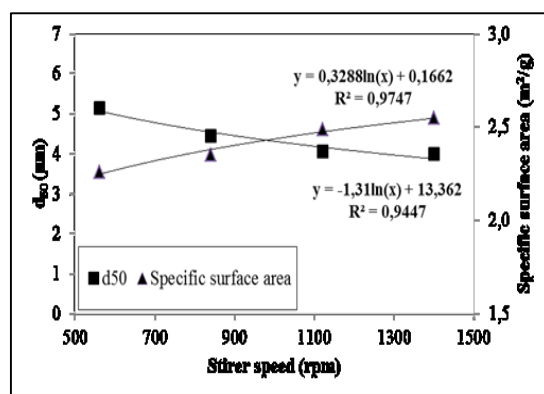


Figure 3. The influences of four different stirrer speeds on d_{50} size and specific surface area

The change in the stirrer speed and d_{50} versus the width of particle size distribution (d_{90}/d_{10}) is illustrated in Figure 4. Accordingly, the width of particle size distribution (d_{90}/d_{10}) decreased with increasing stirrer speed. It may also be observed from Figure 4 that the d_{90}/d_{10} ratio decreased as d_{50} decreased. At higher stirrer speeds (1120, 1400 rpm), the d_{90}/d_{10} ratios were close each other, as the changes in the d_{90} and d_{10} values were negligible for these stirrer speeds. In their studies (using wet Drais Mill and dry Sala Agitated Mill), Wang and Forssberg [10] investigated the effects of stirring speed on the Rosin-Rammler-Bennett modulus and reported that the Rosin-Rammler-Bennett modulus of the products increased as the stirring speed increased.

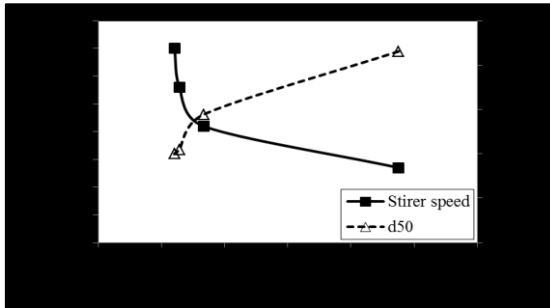


Figure 4. Stirrer speed and d_{50} size as a function of the d_{90}/d_{10} ratio

As a result of the tests that were performed at different stirrer speeds, 840 rpm was selected to be the optimum stirrer speed.

3.2. The Influences of Ball Filling Ratio

Conventional grinding systems (primarily, tumbling ball mills) have been used for grinding processes for many years, but one of their limitations is the level of media charge that achieves a maximum at a charge volume of approximately 40-50%. On the other hand, stirred ball mills can be driven at media charges of up to 85%. The influences of five different ball filling ratios on the size d_{50} and specific surface area are presented in Figure 5. It may be seen that the d_{50} value decreased (from 6.62 to 3.36 μm) when the ball filling ratios (J) changed from 0.4 to 0.8. In the case that the ball filling ratio in a stirred ball mill is high, a rise in the weight proportion of the balls increases the number of collisions per unit time, and as a result, more energy is transferred to the particles, and the sizes are reduced faster [24]. Orumwense [25] found that a finer product size distribution was obtained by increasing the ball filling ratio from 60% to 80%. Gao et al. [26] and Orumwense [25] also reported that excessively high media filling ratios caused sanding, high wear in the media, liner and stirrer, or excessive energy consumptions.

It is also shown in the figure that, as the ball filling ratio is increased, the specific surface area became larger as expected. Altun et al. [23] found that a change in the ball filling ratio from 30% to 60%

increased the Blaine surface area from 1893 to 2357 cm^2/g . Sivamohan and Vachot [27] also observed similar experimental results.

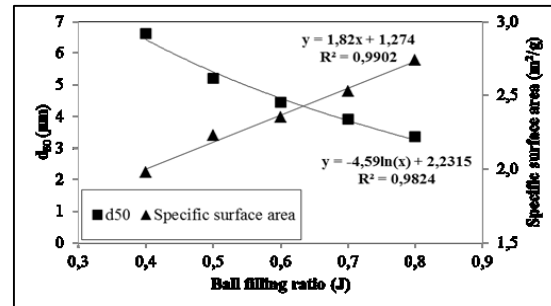


Figure 5. The influences of five different ball filling ratios on d_{50} size and specific surface area

The change in the ball filling ratio and d_{50} versus the width of particle size distribution (d_{90}/d_{10}) is illustrated in Figure 6. Accordingly, the width of particle size distribution (d_{90}/d_{10}) decreased with increasing ball filling ratio. It may also be observed from Figure 6 that the d_{90}/d_{10} ratio decreased as d_{50} decreased.

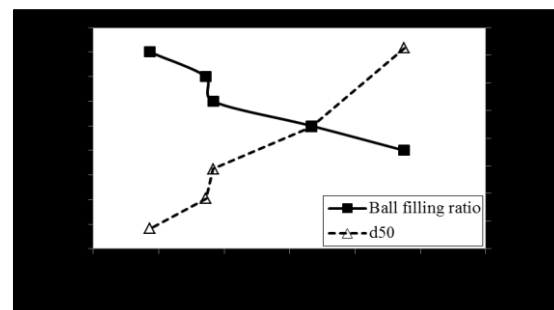


Figure 6. Ball filling ratio and d_{50} size as a function of the d_{90}/d_{10} ratio

An excessively high ball filling ratio in stirred mills causes excessive energy consumption and wear. By considering this fact, $J=0.70$ was chosen as the optimum value.

3.3. The Influences of Powder Filling Ratio

Powder filling or material hold-up is one of the important milling parameters which affect grinding

performance (specific energy consumption, product fineness and specific surface area). In open- or closed-circuit grinding, the amount of material that is held up in the mill changes based on feed rate. In other words, the higher the feed rate is, the higher is the amount of material in the mill [28]. The influences of five different powder filling ratios on the size d_{50} and specific surface area are presented in Figure 7. It is shown that the product fineness decreased with increasing powder filling ratios (f_c). In other words, the powder filling ratio was inversely proportional to the product size. In previous studies, the effect of interstitial filling ratio (U) was investigated in dry feldspar and mica grinding in a stirred ball mill by the author of this study [29–31], and similar results were observed.

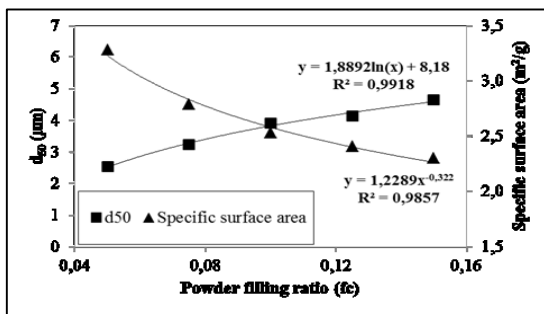


Figure 7. The influences of five different powder filling ratios on d_{50} size and specific surface area

Figure 7 also shows that an increase in the powder filling ratio from 0.05 to 0.15 reduced specific surface area from 3.28 to 2.30 m^2/g . For limestone ground using a stirred ball mill (MaxxMill), Wang and Forssberg [21] concluded that the specific surface area increased from 2.5 to 3.5 m^2/g by decreasing the feed rate from 700 to 300 kg/h at a given rotational speed of stirrer (350 rpm) and given bead sizes (5-7 mm).

The change in the powder filling ratio and d_{50} versus the width of particle size distribution (d_{90}/d_{10}) is illustrated in Figure 8. Accordingly, the width of particle size distribution (d_{90}/d_{10}) increased with increasing powder filling ratio. It may also be observed from the figure that the d_{90}/d_{10} ratio decreased as d_{50} decreased.

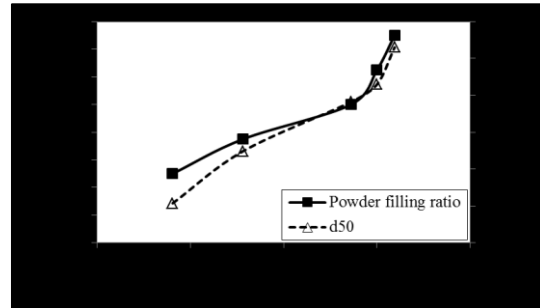


Figure 8. Powder filling ratio and d_{50} size as a function of the d_{90}/d_{10} ratio

A finer d_{50} value and a larger surface area were obtained at the powder filling ratio of $f_c=0.050$ with a lower capacity. However, as calcite is a material that is easy to grind, the powder filling ratio of $f_c = 0.100$ was chosen as the optimum value.

3.4. The Influences of Solid Ratio

The solid ratio is a highly important factor of milling for assessing operations of wet grinding due to its direct effect on the fineness of the ground product and operating energy consumption [4]. Since the fineness of the product increases significantly with grinding time in processes of wet ultrafine grinding that are represented by a very fine particle product size and a high solid ratio, the characteristics of surfaces tend to prevail in the system. Inter-particle forces like van der Waals forces and electrostatic forces result in agglomeration and aggregation [32,33]. The influences of four different solid ratios (wt.%) on d_{50} size and specific surface area are presented in Figure 9. Accordingly, the d_{50} values slightly increased (from 3 to 4.15 μm) when the solid ratio increased from 35% to 80%. The finest product size was obtained with the pulp with a low solid ratio (35%), while no significant change in the product size was observed in the range of 65 to 80% (change in d_{50} from 4 to 4.15 μm). The decrease in grinding efficiency at higher solid ratios may be attributed to the viscosity of the pulp based only upon observation (Figure 10). This result was obtained is similar to the previous study that reported by Dikmen [28] in copper grinding tests.

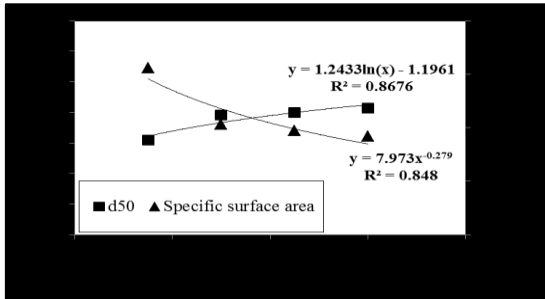


Figure 9. The influences of four different solid ratios on d₅₀ size and specific surface area

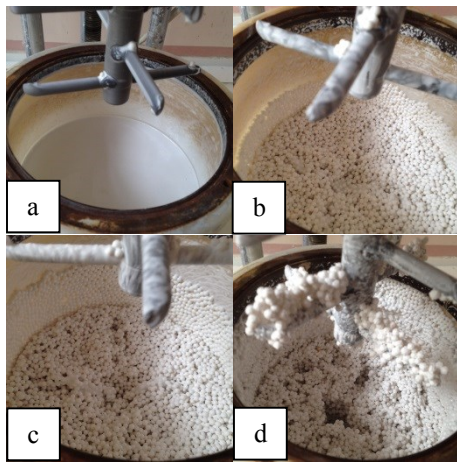


Figure 10. Images of wet grinding of calcite at different solid ratios (a: 35%, b: 50%, c: 65%, d: 80%)

Figure 9 also shows that the specific surface area decreased (from 3.06 to 2.42 m²/g) while the solid ratio increased.

The change in the solid ratio and d₅₀ versus the width of particle size distribution (d₉₀/d₁₀) is illustrated in Figure 11. Accordingly, the width of particle size distribution (d₉₀/d₁₀) was negligible at the solid ratios of 50-65-80%, whereas the width increased at the solid ratio of 35%. These results may be explained in a way by the fact that the change of d₉₀ and d₁₀ values at the solid ratios of 50-65-80% were similar, resulting in no significant change in the d₉₀/d₁₀ ratio. On the other hand, the d₁₀ value decreased faster than the d₉₀ value, and therefore, the d₉₀/d₁₀ ratio increased at the solid

ratio of 35%. Similar interpretations could also be applied for the other results (d₅₀ versus the width of particle size distribution d₉₀/d₁₀). Nettet et al. [16] found that the width of particle size distribution (d₈₀/d₂₀) will be influenced by the media size and to a lesser extent by the media and slurry densities.

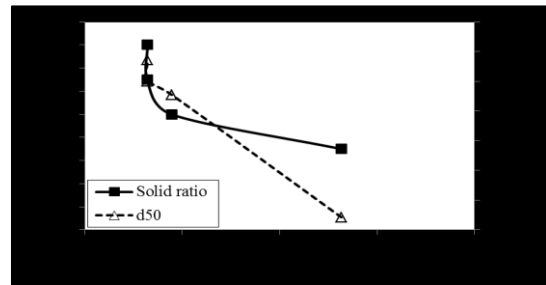


Figure 11. Solid ratio and d₅₀ size as a function of the d₉₀/d₁₀ ratio

As a result of the tests performed at different solid ratios, 50% was selected as the optimum solid ratio.

3.5. The Influences of Grinding Time

For comminution of a batch or charge respectively, namely discontinuous or batch process, circuit operation or pendulum operation, the grinding time is an important indicator for the process of comminution [20]. The influences of five different grinding times (min) on d₅₀ size and specific surface area are presented in Figure 12.

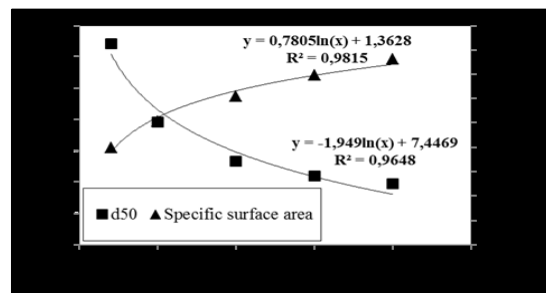


Figure 12. The influences of five different grinding times on d₅₀ size and specific surface area

As the grinding time is increased, as expected, the d₅₀ value decreased, resulting in an increase in the

specific surface area. The finest product size and specific surface area were obtained at 20 min (with d_{50} of 1.95 μm and surface area of 3.81 m^2/g). Orumwense [25] investigated the effects of grinding time (number of passes) and found that increased grinding time made the product size distribution progressively finer. In order to examine the effect of grinding time on the specific surface area, Sivamohan and Vachot [27] tested the effects of number of passes on wollastonite specific surface area using a Drais mill. They found that, by increasing the number of passes, finer particles were obtained. Toraman and Katircioglu [34] also observed similar findings in their studies.

The change in the grinding time and d_{50} versus the width of particle size distribution (d_{90}/d_{10}) is illustrated in Figure 13. Accordingly, the width of particle size distribution (d_{90}/d_{10}) decreased with increasing grinding time. It may also be understood from Figure 13 that the d_{90}/d_{10} ratio decreased as d_{50} decreased. Toraman [35], in their batch stirred mill studies, examined the influences of grinding time on the width of product size distribution (d_{50}/d_{20} , d_{80}/d_{20} , d_{90}/d_{10} and $(d_{90}-d_{10})/d_{50}$). They found that the product size distribution became gradually narrower when the grinding time was increased.

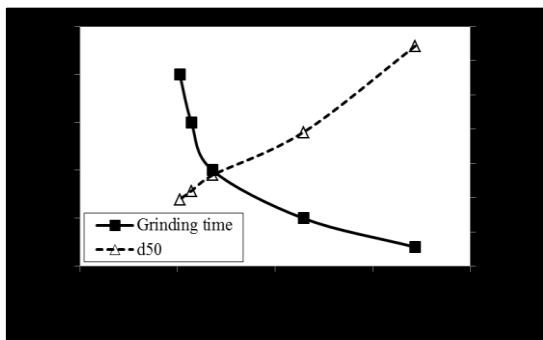


Figure 13. Grinding time and d_{50} size as a function of the d_{90}/d_{10} ratio

As a summary of the results, wet-stirred milling is an effective process for production of ultra-fine calcite particles, and this may be understood by the comparison of the finest product and the feed (Table 3).

Table 3. The comparison of the finest product and the feed

	$d_{50}, \mu\text{m}$	Specific surface area, m^2/g	d_{90}/d_{10}	d_{80}/d_{20}	$(d_{90}-d_{10})/d_{50}$	d_{50}/d_{20}
Feed	41.49	1.03	115.30	19.27	5.18	6.02
Product	1.95	3.81	5.11	2.96	1.73	1.77

3.6. Width of Particle Size Distribution

Particle size parameters (d_{10} , d_{20} , d_{50} , d_{80} and d_{90}) are a valuable indicator of grinding quality and performance. Nevertheless, all these parameters related to particle size have varying importance in different industries. The suitable method of assessing the representation of the width of particle size distribution will thus be determined by the field of application [11]. Figure 14 shows the variations in the ratios of d_{90}/d_{10} , d_{80}/d_{20} , $(d_{90}-d_{10})/d_{50}$ and d_{50}/d_{20} depending on the product sizes (d_{50}) determined for all the tested grinding conditions. In terms of a general reflection on the figures, the width of particle size distribution decreased with decreasing product size for all the tested conditions of grinding. It may be seen in Figures 14a and 14b that the ratios of d_{90}/d_{10} and d_{80}/d_{20} decrease with decreasing product size. In their Pilot Tower Mill and Sala Agitated Mill studies, Jankovic and Sinclair [11] observed that the width of particle size distribution (d_{80}/d_{20}) decreased as the sizes of the product (d_{80}) for both calcite and silica decreased. They also observed in a laboratory Pin Mill batch zinc concentrate grinding study that the d_{80}/d_{20} ratio decreased by decreasing d_{80} (in the case of products finer than 25 μm d_{80}). A similar tendency was observed in quartz grinding tests (regarding the effects of milling type and ball size diameter on the width of particle size distribution) with a tumbling ball mill [18]. There is a small difference between the d_{90}/d_{10} and d_{80}/d_{20} ratios. The d_{90}/d_{10} ratio has a wider range in comparison to that of the ratio d_{80}/d_{20} . Figure 14c shows that the $(d_{90}-d_{10})/d_{50}$ ratio was constant for the product sizes between 3 and 5 μm , and it decreased when the product size became finer than 3 μm . An explanation for the result may be that the rates of change at the values of d_{90} and d_{50} were alike, resulting in no significant change in the ratio of $(d_{90}-d_{10})/d_{50}$, as the effect of d_{10} has a negligible. A similar outcome was also

observed in other experimental studies with used different materials and mills [11,15,18]. As shown in Fig. 14d, the d_{50}/d_{20} ratio decreases with decreasing product size. Werner [17] stated that a d_{50}/d_{20} ratio greater than 2 is explained as “broad,” and ratios of lower than 2 are described as “narrow” or “steep”. In this study, the d_{50}/d_{20} ratio appeared to have a factor of < 2 when the product size became finer than $3 \mu\text{m}$.

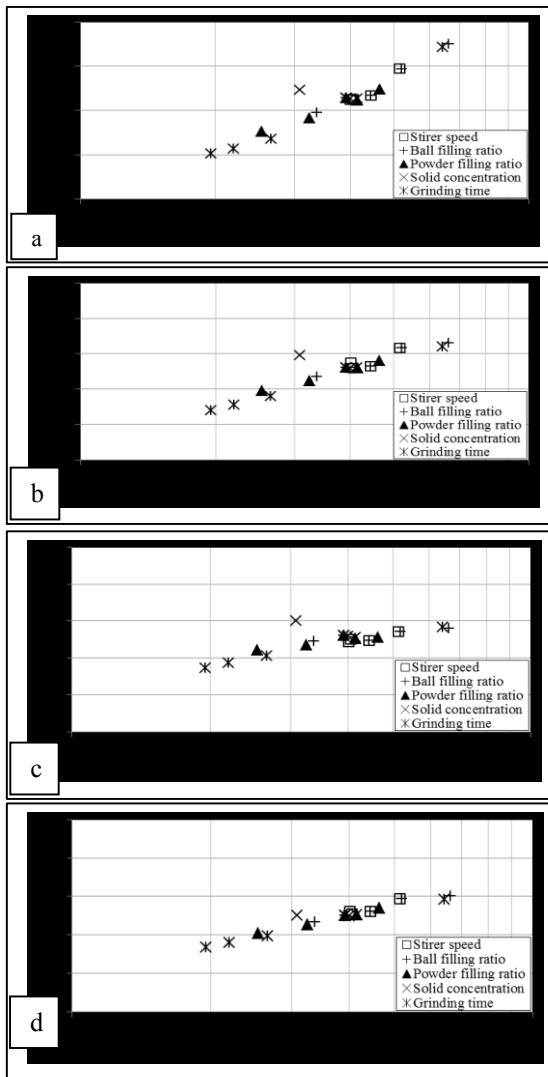


Figure 14. The relationship between the width of particle size distribution (d_{90}/d_{10} , d_{50}/d_{20} , $(d_{90}-d_{10})/d_{50}$ and d_{80}/d_{20}) and the product size (d_{50})

4. CONCLUSIONS

Within the scope of the study, a series of batch grinding tests was performed using calcite powder (CaCO_3) in a laboratory-scale vertical stirred ball mill. The influences of various operating parameters such as stirrer speed (rpm), ball filling ratio (J), powder filling ratio (fc), solid ratio (wt.%) and grinding time on product size (d_{50}), specific surface area (m^2/g) and width of particle size distribution were studied under wet conditions.

- The optimum grinding test conditions were determined to be as follows: stirrer speed=840 rpm, $J=0.70$, $fc=0.100$, solid ratio=50% and grinding time=20 min.

- The grinding test results pointed out that stirrer speed, ball filling ratio and grinding time were directly proportional whereas powder filling ratio and solid ratio were inversely proportional to product fineness and specific surface area.

- The d_{90}/d_{10} ratio that was used to evaluate the width of particle size distribution decreased with decreasing product size for each operating parameter that was tested (excluding solid ratio).

- For all grinding conditions that were tested, the width of particle size distribution decreased with decreasing product size, and the $(d_{90}-d_{10})/d_{50}$ ratio remained constant at the product sizes between 3 and $5 \mu\text{m}$ and decreased as the product size went below $3 \mu\text{m}$, while the d_{50}/d_{20} ratio had a factor of < 2 when the product size became finer than $3 \mu\text{m}$.

5. ACKNOWLEDGMENT

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