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## Influences of operating parameters on dry ball mill performance

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**Abstract:** The paper is aimed to investigate the influence of operating parameters on dry fine grinding of calcite in a laboratory scale conventional ball mill. Within the context, the influence of operating parameters such as mill speed, ball filling ratio, ball size distribution, powder filling ratio, grinding aid dosage and grinding time were studied. The results of grinding tests were evaluated based on the product particle size ( $d_{50}$ ,  $d_{80}$ ) and surface area ( $m^2/kg$ ). As a result of this study, optimum grinding test conditions determined to be 70% of  $N_c$ ,  $J=0.35$  for ball filling ratio, 40 mm (10%), 32 mm (10%), 20 mm (40%), 12 mm (40%) for ball size distribution,  $f_c=0.125$  for powder filling ratio, 2000 g/Mg for grinding aid dosage and 60 min for grinding time. After determining optimum grinding conditions, the influence of grinding aid dosage on powder fluidity was determined. The use of 2000 g/Mg grinding aid dosage had a greater fluidizing effect compared to the other dosages and no aid condition (0 g/Mg). The influence of grinding aid on dry fine grinding of calcite was also examined by Fourier Transform Infrared Spectroscopy (FTIR). FTIR measurements indicated that grinding aid was adsorbed on the ground calcite particles surface.

**Keywords:** conventional ball mill, particle size, surface area, grinding aid, fluidity index, Fourier Transform Infrared Spectroscopy

### 1. Introduction

In recent years, the consumption of industrial minerals in filling and coating applications has increased substantially. Calcite is one of filler materials, which is most commonly and permanently used in the plastic, paint and paper industries. Turkish calcites, in particular, are superior in terms of their quality and reserves. Furthermore, they have a number of significant competitive advantages, for example, high whiteness degree, high  $CaCO_3$  ratio, and very low percentage of iron and silica in impurities. Micronized calcite can be used both directly and in a coated form in the specified sectors. Generally, the milling of calcite is performed in a conventional tumbling mill or stirred ball mill in appropriate fine/ultrafine size ranges for obtaining the desired characteristics of the final product.

Ball mills represent the most widely used and functional kind of a tumbling mill. They are exceptional in terms of their ability to function in various conditions and geometries. Ball mills have been widely utilized in primary, secondary, tertiary and regrind practices for over a hundred years (Napier-Munn et al., 1996). However, ball mills are usually the largest consumers of energy. The cost of energy for grinding is often a determining factor in the economic viability of a mineral activity (King, 1994). Therefore, their efficient use has important performance and cost implications. Researchers have been trying to enhance the comminution efficiency and performance of conventional ball mills for many years. However, it is still required to possess better knowledge of the impacts of mill operating variables in order to achieve the optimum performance (Erdem and Ergün, 2009).

On the other hand, the determination of operational conditions in tumbling milling is usually performed experimentally. For the purpose of identifying the effective operational conditions including media filling, media size, mill speed, and mill diameter, significant effort and time is required (Kano et al., 2000). However, operating parameters are critical indicators influencing the performance (energy efficiency, fineness, surface area, capacity, fluidity, etc.) of a ball mill.

This experimental study is aimed to examine the influence of various operating parameters such as mill speed (% of  $N_c$ ), ball filling ratio ( $J$ ), ball size distribution (%), powder filling ratio ( $f_c$ ), grinding aid dosage (g/Mg) and grinding time (min) on the dry fine grinding of calcite using a laboratory batch scale conventional ball mill. The evaluation of the results of grinding tests was performed based on the product particle size ( $d_{50}$ ,  $d_{80}$ ) and surface area ( $m^2/kg$ ). The influences of a grinding aid on the dry fine grinding of calcite were also examined by the fluidity test and Fourier Transform Infrared Spectroscopy (FTIR).

## 2. Materials and Methods

### 2.1 Materials

The feed material used in grinding tests was calcite ( $CaCO_3$ ) obtained from Nigtas Company (Nigde, Turkey) with the density of  $2.70\text{ g/cm}^3$ . The chemical properties (according to X-ray fluorescence) of the feed material are given in Table 1. The feed material was prepared to the top size of 2.36 mm by using a laboratory scale jaw and roller crusher. The size distribution of the feed material as defined from dry sieving is presented in Fig. 1.

Table 1. Chemical composition of the feed material (wt %)

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

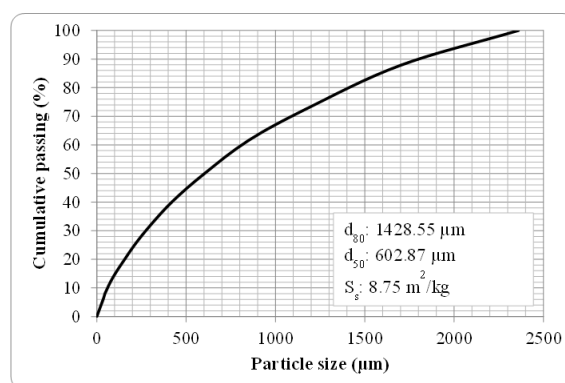


Fig. 1. The size distribution of the feed material

### 2.2. Methods

#### 2.2.1 Conventional ball mill

A laboratory batch scale conventional ball mill which is made of stainless steel with the internal dimensions of  $200 \times 200$  mm and the volume of  $6283\text{ cm}^3$  was utilized in grinding tests. There is no lifter design in the grinding tank. The mill is driven by a  $0.37\text{ kW}$  variable speed motor. Stainless steel cylpebs (having a slightly tapered shape) with the density of  $7.65\text{ g/cm}^3$  and with four different diameters ( $40 \times 40$ - $32 \times 32$ - $20 \times 20$ - $12 \times 12$  mm) represented the grinding media (Fig. 2).

In the mineral sector, grinding balls and rods are commonly utilized as grinding media. However, slightly tapered cylindrical grinding media called cylpebs are also utilized in mineral processing plants. Due to their shape and geometry, cylpebs have some advantages, such as a larger surface area and higher bulk density, in comparison with balls of identical mass and size. Cylpebs of the same diameter and length have 14.5% larger surface area when compared to balls of the equal mass, and 9% higher bulk density when compared to steel balls, or 12% higher in comparison with cast balls. Accordingly, for a specific charge volume, there is approximately 25% more grinding media surface area for size reduction in case of charging with cylpebs (Shi, 2004). Therefore, this experimental study focuses on investigating the influences of various operating parameters on the dry fine grinding of calcite using cylpebs.



Fig. 2. Laboratory ball mill and cylpebs used in the tests

### 2.2.2 The milling conditions

The influences of operating parameters such as mill speed (% of  $N_c$ ), ball filling ratio ( $J$ ), powder filling ratio ( $fc$ ), ball size distribution (%), grinding aid dosage (g/Mg) and grinding time (min) were investigated in this study. The dry grinding test conditions are outlined in Table 2. The grinding tests were conducted as a batch process; after the completion of every grinding test, the mill content was discharged, and the balls were separated from the products by dry sieving.

The commercial liquid grinding aid (the mixture of 80% glycerin – 20% ethanediol) was utilized, and the effect of dosage was investigated in grinding tests. The FTIR spectra of the commercial liquid grinding aid, product (ground calcite with 2000 g/Mg grinding aid dosage for 60 min) and no aid condition (ground calcite without grinding aid for 60 min – 0 g/Mg) were characterized using a Perkin Elmer 2000 spectrometer in the wavelength range of 400-4000  $\text{cm}^{-1}$  equipped with an attenuated total reflectance (ATR) unit (Fig. 11).

### 2.2.3 Determination of particle size ( $d_{50}$ , $d_{80}$ ) and surface area ( $\text{m}^2/\text{kg}$ )

In the studies conducted in the laboratory environment, the particle size of products ( $d_{50}$ ,  $d_{80}$ ) was analyzed from 2360  $\mu\text{m}$  to 0.010  $\mu\text{m}$  by employing dry sieving (a vibrating sieve shaker) and laser diffraction methods (Malvern 2000 Ver. 2.00 with Hydro 2000 MU).

Some simple models, which were stated to be successfully employed in the sector, were developed as a result of the previous studies on the determination of the surface area from the size distribution data of cement (Kuhlmann et al., 1985; Sumner et al., 1989; Zhang and Napier-Munn, 1995).

In this study, the surface area of the products ( $\text{m}^2/\text{kg}$ ) was calculated by the following equation:

$$S_s = \frac{6}{\rho} \sum_{i=1}^n \frac{w_i}{x_i} \quad (1)$$

where  $w_i$  refers to the weight percentage in size fraction  $i$ ,  $x_i$  refers to the harmonic mean size of particle size fraction  $i$  (cm),  $\rho$  refers to the density of the material ( $\text{kg}/\text{m}^3$ ), and  $n$  refers to the number of size fractions (Zhang and Napier-Munn, 1995).

### 2.2.4 Definition of powder fluidity (fluidity index)

After determining the optimum grinding conditions, the influences of grinding aid dosage on powder fluidity were determined. 200 g of the sample was sieved on a 75  $\mu\text{m}$  vibrating sieve as a function of time in order to evaluate the powder fluidity. A fluidity index was then calculated as follows:

$$\frac{P}{P_{max}} = \frac{bt}{1+bt} \text{ or } \frac{t}{P} = \frac{1}{bP_{max}} + \frac{1}{P_{max}} \quad (2)$$

where  $P$  is the amount of calcite passing through the sieve at time  $t$ ,  $P_{max}$  is the maximum amount passing, and  $b$  (a relationship between the fluidity properties of the bulk material and its agglomeration tendency) is the fluidity index (Jolicoeur et al., 2007).

Table 2. Test conditions for dry grinding of calcite

**Mill speed tests**

Parameters	Variable
Mill Speed (% of $N_c$ )	60, 65, 70, 80, 85
Ball filling ratio ( $J$ )	0.35
Ball size distribution of 40, 32, 20, 12 mm (%)	30-30-20-20 (3 <sup>rd</sup> group)
Powder filling ratio ( $f_c$ ), ( $U$ )	0.125, 1.00
Grinding aid dosage (g/Mg)	0
Grinding time (min)	10

**Ball filling ratio tests**

Parameters	Variable
Mill Speed (% of $N_c$ )	70
Ball filling ratio ( $J$ )	0.20, 0.30, 0.35, 0.40, 0.45
Ball size distribution of 40, 32, 20, 12 mm (%)	30-30-20-20 (3 <sup>rd</sup> group)
Powder filling ratio ( $f_c$ ), ( $U$ )	0.125, 1.00
Grinding aid dosage (g/Mg)	0
Grinding time (min)	10

**Ball size distribution tests**

Parameters	Variable												
Mill Speed (% of $N_c$ )	70												
Ball filling ratio ( $J$ )	0.35												
Ball size distribution of 40, 32, 20, 12 mm (%)	<table border="0"> <tr> <td>1<sup>st</sup> group</td> <td>50-50-0-0</td> </tr> <tr> <td>2<sup>nd</sup> group</td> <td>40-40-10-10</td> </tr> <tr> <td>3<sup>rd</sup> group</td> <td>30-30-20-20</td> </tr> <tr> <td>4<sup>th</sup> group</td> <td>20-20-30-30</td> </tr> <tr> <td>5<sup>th</sup> group</td> <td>10-10-40-40</td> </tr> <tr> <td>6<sup>th</sup> group</td> <td>0-0-50-50</td> </tr> </table>	1 <sup>st</sup> group	50-50-0-0	2 <sup>nd</sup> group	40-40-10-10	3 <sup>rd</sup> group	30-30-20-20	4 <sup>th</sup> group	20-20-30-30	5 <sup>th</sup> group	10-10-40-40	6 <sup>th</sup> group	0-0-50-50
1 <sup>st</sup> group	50-50-0-0												
2 <sup>nd</sup> group	40-40-10-10												
3 <sup>rd</sup> group	30-30-20-20												
4 <sup>th</sup> group	20-20-30-30												
5 <sup>th</sup> group	10-10-40-40												
6 <sup>th</sup> group	0-0-50-50												
Powder filling ratio ( $f_c$ ), ( $U$ )	0.125, 1.00												
Grinding aid dosage (g/Mg)	0												
Grinding time (min)	10												

**Powder filling ratio tests**

Parameters	Variable
Mill Speed (% of $N_c$ )	70
Ball filling ratio ( $J$ )	0.35
Ball size distribution of 40, 32, 20, 12 mm (%)	10-10-40-40 (5 <sup>th</sup> group)
Powder filling ratio ( $f_c$ ), ( $U$ )	0.100, 0.125, 0.150, 0.200
Interstitial filling ratio ( $U$ )	0.80, 1.00, 1.20, 1.60
Grinding aid dosage (g/Mg)	0
Grinding time (min)	10

**Grinding aid dosage and time tests**

Parameters	Variable
Mill Speed (% of $N_c$ )	70
Ball filling ratio ( $J$ )	0.35
Ball size distribution of 40, 32, 20, 12 mm (%)	10-10-40-40 (5 <sup>th</sup> group)
Powder filling ratio ( $f_c$ ), ( $U$ )	0.125, 1.00
Grinding aid dosage (g/Mg)	0-500-1000-2000-4000
Grinding time (min)	10-20-30-60

$$N_c = 42.3/\sqrt{(D - d)} \tag{3}$$

$$J = \frac{\text{mass of balls / ball density}}{\text{mill volume}} \times \frac{1.0}{0.64} \tag{4}$$

$$f_c = \frac{\text{mass of powder / powder density}}{\text{mill volume}} \times \frac{1.0}{0.64} \tag{5}$$

$$U = \frac{f_c}{0.36J} \tag{6}$$

### 3. Results and discussion

#### 3.1 The influences of mill speed

Ball mills are frequently operated at higher speeds, therefore, the balls cataract and impact on the particles. There is an increase in the work input to a mill in relation to the speed, and ball mills are operated at a maximum high speed without centrifuging. As it is known, the mill speed of a ball mill is important in ensuring the efficient mill operation. Test results of investigating the influence of mill speed (% of  $N_c$ ) are given in Fig. 3. In Fig. 3, the change in mill speed from 60% to 70% decreases the  $d_{50}$  and  $d_{80}$  values; however, further mill speed increase also elevates the  $d_{50}$  and  $d_{80}$  values. It is also understood from the Fig. 3 that similar discussions could also be applied to surface area results except for the range of 60-70%. The possible explanation is that cataracting at 70% of  $N_c$  converted the potential energy of the medium into kinetic energy of impact on the toe of the charge and as much very fine material was not produced as in case of abrasive grinding by cascading at lower speeds. Even faster speed (0.85 of  $N_c$ ) resulted in the media centrifuging inside the mill (the centrifuging layer gets thicker for smaller media as the speed increases after a certain point), and virtually less milling or movement of the media or product occurred.

Fig. 3 also indicates that the change in the surface area at 70-80-85% of  $N_c$  is negligible due to the fact that the surface area includes not only  $d_{50}$  but also all size components. As a result of the tests performed at different mill speeds, 70% of  $N_c$  was selected to be the optimum mill speed and agreed with the general rule that optimum grinding occurs at a certain speed, which is lower than the critical speed.

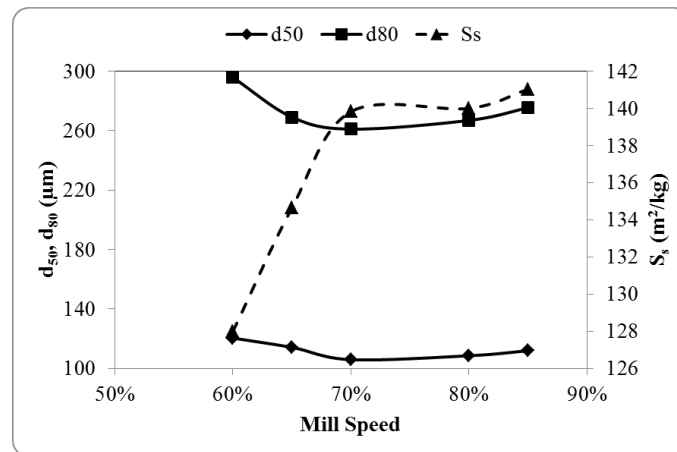


Fig. 3. The results of mill speed tests

#### 3.2 The influence of ball filling ratio

Media filling represents one of the most significant operating parameters directly affecting the product fineness. Furthermore, mill filling has the major impact on the power draw and the specific energy consumption. More energy is needed when there is an increase of the mass inside the mill. On the other hand, the charge volume makes up approximately 40-50% of the ball mill's internal volume. An increase in the energy input to a mill is observed with the ball charge, and its maximum value is achieved at a charge volume of about 50% (Wills and Napier-Munn, 2006).

Fig. 4 illustrates the influence of the ball filling ratio ( $J$ ) on the surface area and particle size ( $d_{50}$ ,  $d_{80}$ ). It is observed from the figure that the  $d_{50}$  and  $d_{80}$  values decrease with an increase of ball filling ratio from 0.20 to 0.35 and then increase at 0.40-0.45. It is also understood from the figure that the highest

surface area was obtained at the ball filling ratio of 0.35. These results can be explained by the fact that collision spaces between the balls were filled and higher breakage rates were acquired at the filling ratio of 0.35.  $J > 0.35$  increased the mill hold-up. However, an increase in the breakage rates was not ensured due to the fact that the collisions zones had been already saturated. Underfilling ( $J < 0.25$ ) led to the deadening of the collisions by powder cushioning, the ball-powder bed expanded to give poor ball-ball powder nipping collisions and a decrease in milling performance was observed. Deniz (2012) in his dry ball mill studies investigated the impacts of media filling on the kinetic breakage parameters of a barite sample, and he found out that the filling ratio of 0.35 was a suitable value for barite grinding. Gupta and Yan (2006) also indicated that 70-80 % of the critical speed and a ball charge of 35-45% of the mill volume would be the optimum conditions for grinding and the speed of rotation determines the position of the charge in a mill. As a result of the tests performed at different ball filling ratios,  $J=0.35$  was selected to be the optimum ball filling ratio to obtain a finer particle size ( $d_{50}$ ,  $d_{80}$ ) and a higher surface area.

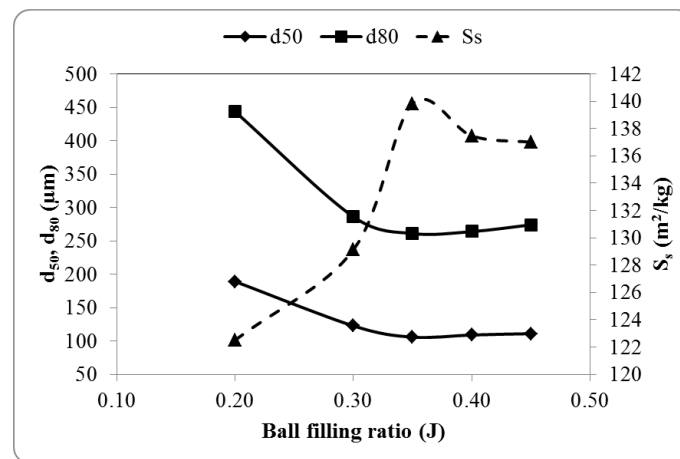


Fig. 4. The results of ball filling ratio tests

### 3.3 The influence of ball size distribution

Ball size represents an important parameter, which affects the performance and capacity of a ball mill. With an increase in the size of balls, the balls will be able to crash large particles, but it should be noted that an increase in the size reduces the number of balls, which leads to a reduction in the grinding rate. Therefore, the grinding efficiency decreases. On the other hand, a decrease in the ball size leads to the increased number of balls, but under such a condition, large particles would leave the mill without being ground (Bond, 1958). In the literature, there are studies conducted on the selection of an optimum ball size (Tarjan, 1981; Duda, W.H., 1985; Ucurum et al., 2015; Oksuzoglu and Ucurum, 2016). Various researchers also examined the relationship between  $D_b$  (ball size) and  $X_m$  (particle size at which the highest breakage takes place) (Austin et al., 1984; Napier-Munn et al., 1996; Man, 2000).

Fig. 5 shows the impact of ball size distribution (%) on the particle size ( $d_{50}$ ,  $d_{80}$ ) and surface area. As can be seen, coarser ball loads (the 1<sup>st</sup> and 2<sup>nd</sup> group) were found to be inefficient in breaking particles. Furthermore, a finer ball load (the 6<sup>th</sup> group) has a larger surface area, but it is too weak to break the coarser particles nipped in the feed. The 5<sup>th</sup> group ball size distribution has the best grinding performance ( $d_{50}$ ,  $d_{80}$  and surface area) among the ball size distributions tested. Ucurum et al. (2015) in wet calcite grinding test point out that the best results were obtained at 50% (10 mm), 50% (20 mm), 0% (30 mm), and 0% (40 mm) ball size distribution. Oksuzoglu and Ucurum (2016) in their dry gypsum grinding studies investigated the effect of ball size distribution using spherical balls and found that a finer product size was obtained at 20% (20 mm) - 60% (30 mm) - 20% (40 mm) ball size distribution. Erdem and Ergün (2009) modeled the grinding process in a pilot ball mill and calculated the specific breakage rate. They concluded that coarser grinding media fillings were found to be efficient for coarser feed particles and finer grinding media fillings having larger surface area were efficient for feed particles smaller than 0.212 mm. Various formulae and relationship have been described by the researchers. Nevertheless, there are a lot of factors that influence the selection of an appropriate ball size affecting

feed size distribution, mill diameter, feed hardness, the specific gravity of the ball, ball shape and the surface area of the ball.

As a result of the tests performed at different ball size distributions, the 5<sup>th</sup> group (10% (40 mm) - 10% (32 mm) - 40% (20 mm) - 40% (12 mm)) was selected to be the optimum ball size distribution for these test conditions.

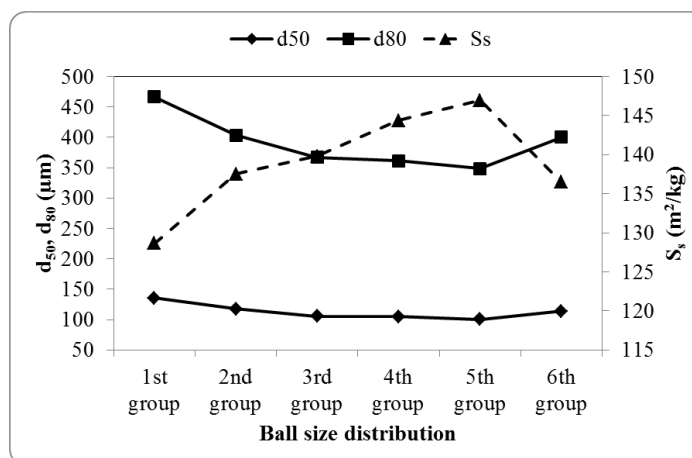


Fig. 5. The results of ball size distribution tests

### 3.4 The influence of powder filling ratio

The operational conditions and the grinding mechanism determine the grinding capacity of a mill. The absolute rate of breakage or the mill capacity is generally presented as the output of the selection function and a powder hold-up, or a fractional volume of powder filling (Deniz, 2011). Underfilling as well as overfilling the mill with powder should be avoided for a specific ball loading. At lower powder filling, a significant part of the energy of tumbling balls becomes involved in the ball to ball contact, which causes a higher wear rate. In the case of overfilling, the deadening of the collision by powder cushioning is observed, there is an expansion of the ball-powder bed which provides poor ball-ball-powder nipping collisions, and there is a decrease in the milling performance. In practice, the preferred ratio of the bulk material charge / the bulk rock charge  $\sim 0.4$  and the volume fraction of voids at rest, occupied by the material, varies between 0.6 and 1.1. In the literature, milling kinetics has been studied as a function of powder filling by various researchers (Shoji et al., 1982; Deniz and Onur, 2002; Tangsathitkulchai, 2003; Deniz, 2011). In the study conducted by Shoji et al. (1982), the impacts of powder filling on grinding were examined for the wet and dry grinding of quartz in a laboratory ball mill. They found the maximum at  $U = 0.83$  for the quartz mineral. In the study of Deniz and Onur (2002), it was implied that an interstitial filling fraction of  $U = 0.4$  represents a good powder-ball loading ratio for pumice. In the study carried out by Tangsathitkulchai (2003), it was indicated that grinding at approximately  $I = 1$  provided the maximum in net mill power. Deniz (2011) also found that the high value of  $U = 1.5-2.0$  is an effective ratio for gypsum. The differences possibly result from different minerals, densities and grindabilities.

The influence of different powder filling ratios ( $f_c$ ) on the  $d_{50}$ ,  $d_{80}$  sizes and specific surface area are illustrated in Fig. 6. It is shown that the  $d_{50}$  and  $d_{80}$  values increase with an increasing powder filling ratio ( $f_c$ ), which results in a decrease of the specific surface area. In addition, the product fineness and the surface area increases linearly with a decreasing powder filling ratios ( $f_c$ ).

Finer  $d_{50}$ ,  $d_{80}$  values and larger surface area were obtained at the powder filling ratio of  $f_c = 0.100$  having a lower capacity and calcite is a material of easy grind. Thus, the powder filling ratio of  $f_c = 0.125$  was selected to be the optimum and agreed with the general rule ( $U = 0.6$  to 1.1).

### 3.5 The influence of grinding aid dosage and time

Grinding aids have been utilized in mineral processing applications for a long time. Chemicals used as grinding aids, particularly in dry grinding processes, lead to a significant increase in energy saving, cement strength, surface area, energy efficiency, size reduction, avoid the agglomerates or aggregates

of ground particles, prevent grinding media coating and enhance material transport (Zheng et al., 1997; Choi et al., 2009; Toprak et al., 2014; Gokcen et al., 2015). The test results of investigating the influences of grinding aid dosage (g/Mg) and time (min) are presented in Fig. 7 and Table 3. Fig. 7a and 7b show that product fineness ( $d_{50}$ ,  $d_{80}$ ) and surface area slightly increase at the grinding aid dosage of 500 g/Mg compared to no aid condition (0 g/Mg) and other grinding aid dosages. The fact that adhesive tendencies between particles would be reduced in the most effective way by grinding aids chemisorbing on solid particles and therefore grinding efficiency would be improved could be the reason for this. Due to the fact that adhesive forces are dependent on the particle size (surface area), the finer the size of the product is, the more noteworthy is the relative improvement in grinding efficiency (Fuerstenau, 1995).

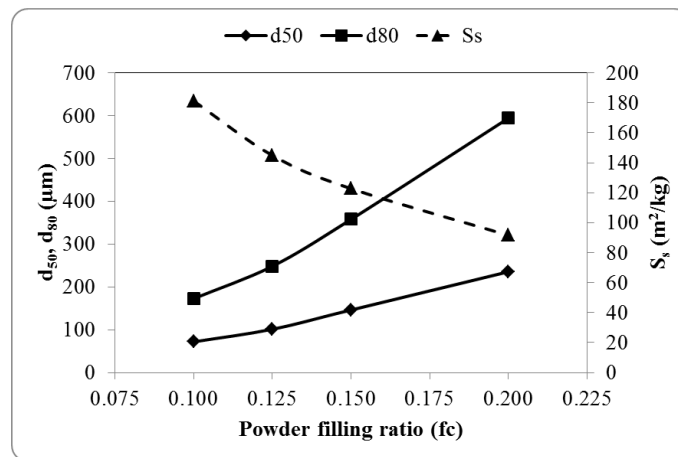


Fig. 6. The results of powder filling ratio tests

Table 3. The variation of mill performance with grinding aid dosages and times

Grinding aid dosage (g/Mg)	10 min			20 min			30 min			60 min		
	$d_{50}$ (µm)	$d_{80}$ (µm)	$S_s$ (m <sup>2</sup> /kg)	$d_{50}$ (µm)	$d_{80}$ (µm)	$S_s$ (m <sup>2</sup> /kg)	$d_{50}$ (µm)	$d_{80}$ (µm)	$S_s$ (m <sup>2</sup> /kg)	$d_{50}$ (µm)	$d_{80}$ (µm)	$S_s$ (m <sup>2</sup> /kg)
No aid	100.3	248.2	139.0	36.0	101.8	270.0	24.6	78.7	354.0	16.0	61.0	478.0
500	99.9	236.5	144.9	34.0	97.7	271.5	23.6	68.1	355.3	14.0	42.9	480.0
1000	108.0	251.6	125.0	37.8	106.3	267.4	21.7	59.8	364.0	13.5	37.1	484.0
2000	124.9	287.2	125.6	41.4	114.6	271.8	24.4	67.4	362.0	13.3	33.7	486.0
4000	131.4	298.9	109.9	56.4	163.7	214.22	27.9	82.5	323.6	13.7	41.0	465.1
Feed	602	1428	8.75	602	1428	8.75	602	1428	8.75	602	1428	8.75

The influences of grinding aid are more significant in the later stages of grinding (30 and 60 min.), as shown in Fig. 7c and 7d. As it can be seen from the figures, finer  $d_{80}$  values were obtained with 24% at 1000 g/Mg (30 min.) and 45% at 2000 g/Mg (60 min.) compared to no aid condition. On the other hand, it is observed that grinding aid is less effective on the  $d_{50}$  values and surface area compared to no aid condition. Upon comparing the results, the use of 1000 g/Mg grinding aid dosage at 30 min grinding time and 2000 g/Mg grinding aid dosage at 60 min grinding time in the mill can indicate enough beneficial effect on the product size ( $d_{50}$ ,  $d_{80}$ ) and surface area (Table 3). In other words, grinding performance (the maximum value of product fineness and surface area) improved and the relevant grinding time became longer as the grinding aid dosage increased. Hasegawa et al. (2001) observed a similar tendency in their quartz grinding tests with a vibration rod mill. They also suggested that the effective grinding dosage increases with the fineness of the material. Additionally, it is observed that the dosage rate of 4000 g/Mg has no considerable effect on grinding performance among the grinding aid dosages for each grinding time tested. This decrease can be explained by the reagglomeration of the particles, which results in negative grinding performance. Toraman (2012) in his dry stirred media mill studies investigated the influence of grinding aid dosage on the product size and surface area. He found out that the increase in grinding aid dosage (from 0.05 to 0.3%) increased product fineness and surface



area, to reach a maximum value (amount of 0.2%), and then to decrease (amount of 0.3%). Oksuzoglu and Ucurum (2016) also observed a similar tendency in their gypsum dry ball milling studies.

3.5.1 Powder fluidity (fluidity index)

The use of grinding aids has influenced material transportation (powder fluidity) in the dry grinding process. The increased powder fluidity ultimately creates a more efficient grinding environment. Within the context, the fluidity index ( $b$ ) is calculated by sieving as a function of time in order to evaluate the powder fluidity. Fig. 8 shows the weight of calcite powder passing through the sieve over time. The fluidity indices were obtained from ground calcite with and without grinding aid at 60 min grinding time, which is illustrated in Fig. 9. As it is understood from the figure, an increase in the fluidity index is observed with an increase in the dosage of grinding aid, reaching a maximum value (at 2000 g/Mg) and then decreasing (at 4000 g/Mg), hence the dosage rates of 2000 g/Mg have lower tendency of forming agglomerates. On the other hand, the decrease in the fluidity index at the dosage rate of 4000 g/Mg explained by the reagglomeration of the particles, which results in negative fluidity performance. The results presented in Fig. 8 prove that the decrease in the fluidity index at the dosage rate of 4000 g/Mg resulted in obtaining less weight of calcite powder passing through the sieve compared to the dosage rates of 1000 and 2000 g/Mg. It can also be observed from Fig. 9 that the calculated fluidity index of all results is higher when compared to no aid condition. As the use of grinding aids disperses the particles and prevents grinding media coating, grinding media transfer the energy more efficiently, thus, material transport and grinding performance improves (Altun et al., 2015). Jolicoeur et al. (2007) tested the effects of glycols and compared the results with triethanolamine and found the fluidity index. They concluded that the calculated fluidity index of triethanolamine was higher when compared to glycols and no aid condition. Altun et al. (2015) studied the influences of grinding aids on cement transportation in a stirred ball mill by calculating the residence time of cement. They found out that triisopropanolamine decreased the residence time of cement from 154 seconds (0 g/Mg) to 101 seconds (700 g/Mg).

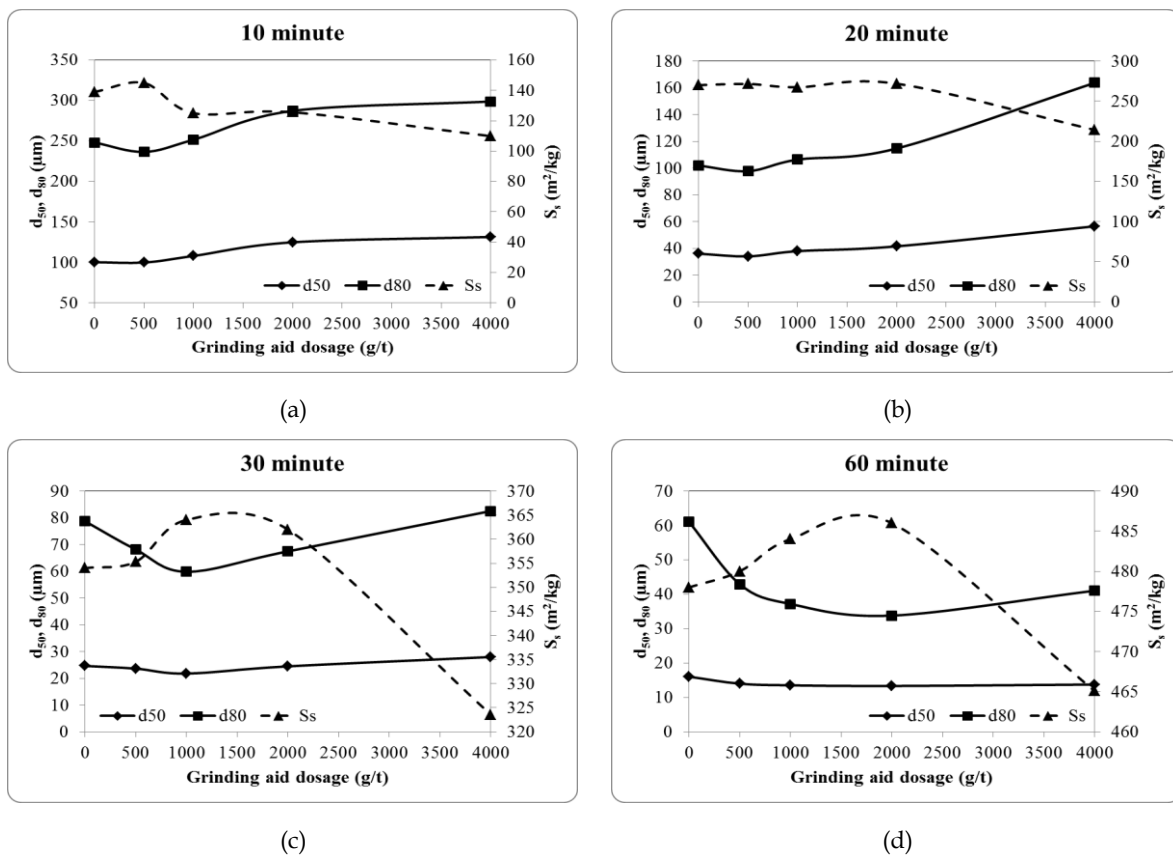


Fig. 7. The results of grinding aid dosage and time tests

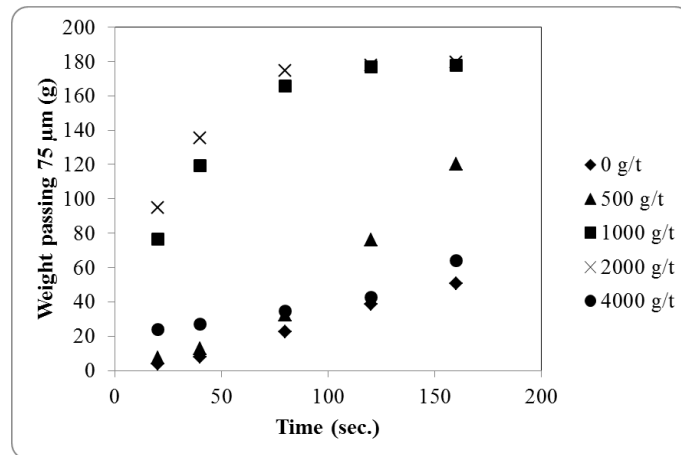


Fig. 8. Influence of grinding aid dosage on powder fluidity as determined by sieving

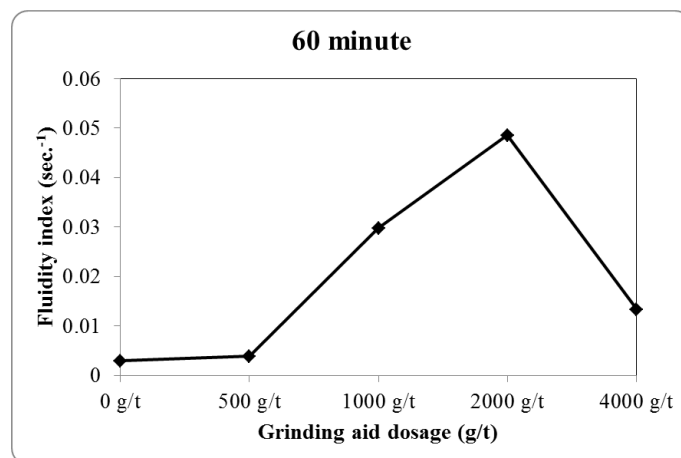


Fig. 9. Fluidity of calcite products at different dosages of grinding aid

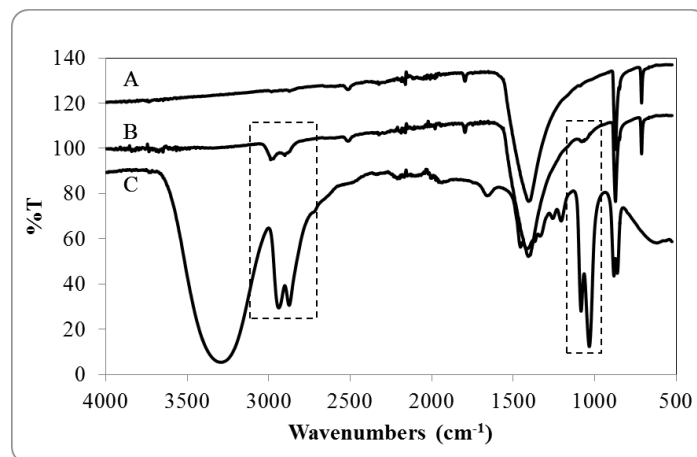


Fig. 10. FTIR spectra of no aid condition (A), product (B) and commercial liquid grinding aid (C)

### 3.5.2 FTIR measurements

The FTIR spectra of the commercial liquid grinding aid (C=mixture of 80% glycerin - 20% ethanediol), product (B=ground calcite with 2000 g/Mg grinding aid dosage for 60 min) and no aid condition (A=ground calcite without grinding aid for 60 min) are presented in Fig. 10. The FTIR spectra in Fig. 10A present the characteristic absorption bands of calcite located in 1394, 872 and 712  $\text{cm}^{-1}$  (Labidi and Djebaili, 2008; Yoğurtcuoğlu and Uçurum, 2011). As can be seen from the FTIR spectra of the liquid grinding aid in Fig. 10C, stretching of ether groups (C-O) appeared at  $\nu=1032\text{-}1083 \text{ cm}^{-1}$ . The

characteristic alkyl (C-H) stretching modes from  $\nu=2800-3000\text{ cm}^{-1}$  were observed. Furthermore, the contribution of a hydroxyl group (broadband) was observed with absorption varying between  $\nu=3000$  and  $3500\text{ cm}^{-1}$  (Mansur et al., 2004). In the infrared spectrogram of the product, the characteristic absorption peaks of C-H were observed at a wavenumber slightly lower than  $3000\text{ cm}^{-1}$ , indicating that the grinding aid was adsorbed on the surface of calcite when compared to the FTIR spectra of no aid condition. Furthermore, the weak absorption peaks at  $\nu=1032$  and  $1083\text{ cm}^{-1}$  were attributed to the C-O stretching vibrations of the grinding aid.

#### 4. Conclusions

In this experimental study, the influence of various operating parameters such as mill speed (% of  $N_c$ ), ball filling ratio ( $J$ ), ball size distribution (%), powder filling ratio ( $f_c$ ), grinding aid dosage (g/Mg) and grinding time (min) on dry calcite grinding were examined using the laboratory batch scale conventional ball mill. The experimental results were evaluated on the basis of product size ( $d_{50}$ ,  $d_{80}$ ) and surface area ( $\text{m}^2/\text{kg}$ ). The optimum grinding conditions of calcite was determined to be as follows; 70% of  $N_c$  for mill speed,  $J=0.35$  for ball filling ratio, 40 mm (10%), 32 mm (10%), 20 mm (40%), 12 mm (40%) for ball size distribution,  $f_c=0.125$  for powder filling ratio, 2000 g/Mg for grinding aid dosage and 60 min for grinding time. These conditions produced the  $d_{50}$  of 13.33 and  $d_{80}$  of 33.79  $\mu\text{m}$  with the surface area of 486  $\text{m}^2/\text{kg}$ , with respect to the feed samples with the  $d_{50}$  of 602 and  $d_{80}$  of 1428  $\mu\text{m}$  ( $S_s= 8.75\text{ m}^2/\text{kg}$ ) obtained by dry grinding in a conventional ball mill with steel cylpebs. The results point out that operating parameters were critical indicators influencing the performance of a ball mill.

After determining the optimum grinding conditions, the influence of grinding aid dosage on powder fluidity were determined. The use of 2000 g/Mg grinding aid dosage has a greater fluidizing effect compared to the other dosages and no aid condition (0 g/Mg). The mechanism of grinding aid on the calcite grinding was also examined by FTIR. FTIR measurements indicated that grinding aid was adsorbed on the ground calcite particle surfaces.

As an overview, conventional ball mills are employed in grinding processes for more than a century because of some advantages. Many studies have been performed to enhance the grinding efficiency and performance of conventional ball mills by various researchers. However, the studies on improving the grinding performance are still ongoing.

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