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Characterization of Surface Roughness of Calcite by BET and Surtronic 3^+ Techniques

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ABSTRACT: Experimental investigations to determine the surface roughness of calcite mineral ground in ball, rod and autogenous mills by BET (Brunauer-Emmett-Teller) and Surtronic 3^+ techniques are presented. The surface roughness values of particles determined by the BET technique were stated in terms of the RBET values by calculating from the measured BET surface area values and varied between 15.98 and 19.98. The surface roughness values of particles in pelleted forms determined by Surtronic 3^+ direct measurement technique were also stated in terms of the R., values and varied between 2.90 and 3.54 um. Considering the surface roughness values of calcite mineral ground in different mills determined by both techniques, the lower roughness values were observed in the autogenously ground particles while rod mill products had higher surface roughness values. Finally, some correlations were made between the calculated surface roughness (RBET) and measured surface roughness ($R_{,L}$) values, in which the R„ values increase with increasing RBET values. The empirical relationship was found as RBET= 6.24 R_a - 2.16.

I INTRODUCTION

Grinding by different mills is very important for the minerals for further processing in mineral processing operations, In fact, it is essential to liberate and increase the surface area of valuable minerals from the gangue minerals. (Frances et al., 2001).

Mechanical grinding causes considerable changes in surface.

The different types of mills can be classified as regards to the main stresses that act on the particles: compression, shear, attrition, impact and internal forces. It is also difficult to discriminate these stresses on the particles, because at least two of them are obviously simultaneously acting in each machine. However, three modes of breakage are usually defined, which are abrasion, chipping and impact breakage (Redner, 1990).

Specific surface area (area per unit mass or volume) is a useful measure of particle size, characterization and roughness. When a clean solid surface is exposed to a gas (e.g.; NT). the gas molecules are attracted to the surface and form adsorbed layers. Under fixed conditions, the extent of adsorption is proportional to the total surface area of the solid (Hogg, 1980; Allen, 1975).

The BET (Brunauer-Emmett-Teller) method measures all the area, which can be reached by the molecules of the gas being used (often N: at liquid N: temperatures) (Adamson, 1976).

At a small enough scale, however, all real surfaces contain some degree of nonuniformity. Because of this, discrepancies between theoretical predictions and experimental observations are frequently attributed to surface roughness effects (Suresh&Walz, 1996).

In general, roughness is confined to changes in the surface representing movements of the surface larger than the interatomic distances (Jaycock& Parfitt, 1981).

Surface roughness is, most likely, due to fluctuations around a smooth and sharp interface, but may also represent a lower free energy state (Szleiferetal., 1986).

Surface roughness is becoming increasingly important applications in many fields (Bennett, 1992).

Roughness of surfaces is of interest to a wide range of researchers in materials science (Lange et. al., 1993). The roughness of the particles can be estimated by the measurement of BET gas adsorption. Once the surface area is measured, roughness of the surface can be characterized.

$$R_BET=A_BET.d.(D/6) \qquad (1)$$

where, ABET is the BET surface area measured, d is the density of solid and D is the particle diameter (Jaycock&Parfitt, 1981).

Surface roughness is known to play an important role in the spreading behavior of liquids and the measurement of the macroscopic contact angle.

The surface roughness measurement method is based on the mechanical sensing of surface topography in combination with electronic amplification of the signal obtained (Rank Precision Industries, 1944). Such measurements represent a highly accurate method (reproducibility within 10 Angstrom of vertical resolution in the latest types of equipment, i.e. Talystep 1 of the Rank-Taylor Hobson Co.) for rapid, non-destructive analysis of the surface topography. The measuring element of the apparatus is essentially the same as the ones found in the pick-up element of present-day gramophones. Vertical displacements, stemming from the sample moving at constant speed along the diamond needle of the sensing element, are amplified electronically. The fact that the actual measuring procedure is fully electronic ensures instant read-out. The main advantage of the method of surface roughness is to be found in the direct mechanical nature of the technique (Deelman, 1977).

The purpose of this study is to determine the surface roughness values of calcite particles as an example mineral to study, using two different methods that are BET gas adsorption and direct measurement by Surtronic 3^{+}, and also investigate and correlate the validity of these two approaches.

2 MATERIALS AND METHOD

2.1 Materials

The mineral used in the grinding test, surface area measurements and surface roughness measurement tests were calcite from Niğde locations of Turkey. This material was prepared by crushing to -20+30 mesh sieve fractions for ball milling, -4+6 mesh sieve fractions for rod milling and -10+1 cm for autogenous milling. The chemical compositions of calcite mineral is given in Table 1. Figure 2 and 3 show the composition of calcite sample used in the experiments by SEM and XRD techniques, respectively.

Table 1. Chemical composition of calcite mineral used in this study.

CaCO,. </K	SiO,. %	MgO. %	R;0,. %
99.14	0.30	0 11	0.25

After grinding for a certain time, the appropriate amounts of fine mill products were saved for further tests such as BET surface area measurements and the direct surface roughness measurements.

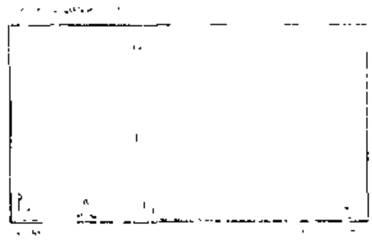


Figure 1. Chemical composition of caicite used in the study by SEM

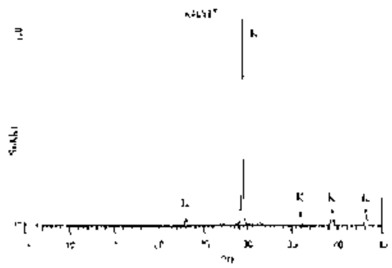


Figure 2. Mıneralogıcal analysis of calcite used in the study by XRD

2.2 *Mills*

The ball milling experiments were performed in a cylindrical mill of 200 cm internal diameter and 5776 cm' volume, using the mixture of steel balls of 30 mm and 26 mm diameters that weight 5475 g. A loading of 20 % of the mill volume filled by the ball bed, a fractional interstitial filling of the bed voids by the dry powder of 0.5 at 75 % of the critical speed were chosen to run the tests. Feed size of the mineral was 374 g for calcite of -800+600 urn.

Sieving schedules were established which gave complete sieving without excessive abrasion from a sieving kinetics study, a required sample taken by cone and quartering technique was wet sieved first and then dry sieved for 10 minutes in a Ro Tap shaker for screen analysis.

A conventional rod mill has been used, with an internal diameter of 200 mm and volume of 8792 cm using a total 22.6 kg rod in different diameters (29. 24 and 19 mm in diameter). Feed size of the mineral for rod milling was -4750+3350 um. The sieving procedures were set for 10 minutes of dry screening following the wet screening on the samples taken by cone and quartering technique.

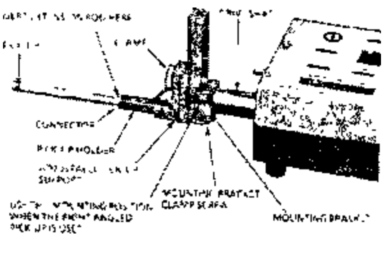
For dry autogenous grinding, a 420x225 mm laboratory scale autogenous mill with 31156 cm volume and rubber lining was employed. The charge as media consisted of 3.0 kg of -80+50 mm pebbles. The mineral Iced size to grind was -10 000+1000 urn that weights 2.0 kg. Sieving schedules was the same as described in the rod milling case.

2.3 The BET surface area measurements

The surface area of particles ground in ball, rod and autogenous mills was measured by using Micromentics Flowsorb II 2300, which employs BET nitrogen adsorption technique. The surface roughness (RBET) of the calcite particles was calculated from the BET measurements by using equation (1). For each mill products three measurements were made and the RBET values determined by taking the average of the three measured values.

*2.4 Direct measurement of surface roughness by Surtronic 3**

In order to determine the surface roughness of particles, a portable stylus-type roughness-measuring instrument, Surtronic 3^+, which has a microprocessor was used (Figure 3). It measures the average roughness (R„) values directly by traversing across the surface of the pellets formed. For each mill products three measurements were made and the R„ values determined by taking the average of those three values.



Figme 1 Siirlionic 3* Instrument used in the direct roughness measurements

3 RESULTS AND DISCUSSION

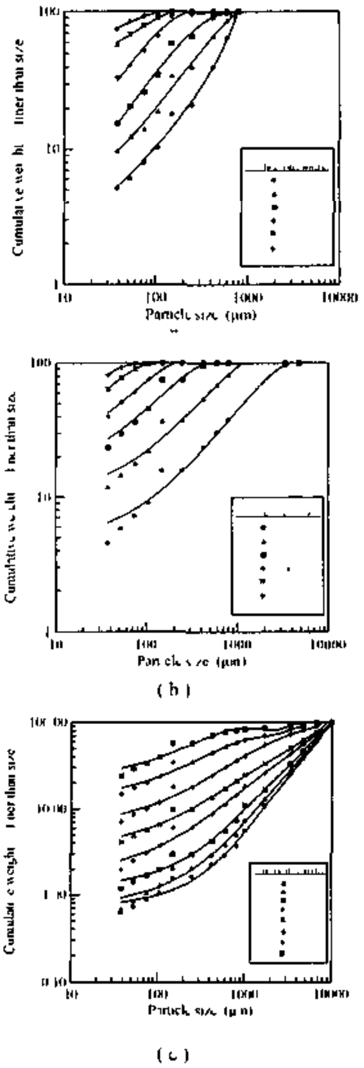
Figure 4 shows the product size distributions of calcite mineral ground dry in ball, rod and autogenous mills. The feed sizes were -800+600um for ball mill, -4750+3350um for rod mill and -80+50 mm of media mineral and -10+1 mm of ore to be ground for autogenous mill. In order to carry out BET surface area measurements and Surtronic 3^+ surface roughness test, the appropriate amounts and size of samples were taken from these grindings; i.e: the grinding time to give 100 % passing at 250 urn was the optimum grinding length to save samples for further tests. These grinding times were 16 minutes for ball and rod mills; while they were 128 minutes for autogenous mill depending on the mineral as whose product size distributions with the grinding times were given in Figure 4 (a) through (c).

Abrasion is the main mechanism of comminution in autogenous mills, while impact crushing predominates in ball and rod mills. Hence, in autogenous grinding liberation and size distribution of the ground product depend on mineralogical properties: a rock with stable grains and weak boundaries gives a steeper size distribution and a better liberation in the autogenous mill than by ball milling (Digre, 1979).

The surface areas of the particles measured by BET nitrogen adsorption technique for these minerals ground in three grinding mills are outlined in Table 2. The calculated surface roughness (RRCT) values using equation (1) are also included in the same Table. Figure 5 shows the bar graphs of the BET surface areas of calcite mineral with the grinding mills employed.

As it is easily seen from Table 2 and Figure 5, the surface roughness (RBET) values are the highest for rod mill product and the lowest for autogenous mill product of calcite mineral. This is due to the breakage characteristics of mineral being ground and the type of mill used for the comminution of the mineral.

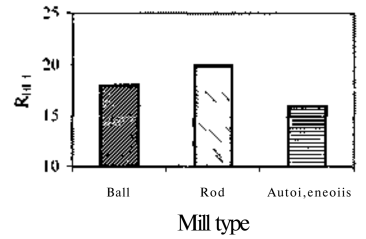
The results of the direct measurement of surface roughness (R„) values of calcite mineral by Surtronic 3^+ instrument are outlined in Table 2 and the comparison of the R„ values determined by Surtronic 3^+ technique is shown in Figure 6 for each mill. The surface roughness (R„) values are the highest for rod mill product and the lowest for autogenous mill product of calcite as in the case of BET measurements. This was also attributed to the breakage characteristics of the calcite mineral and grinding actions of the mills employed.



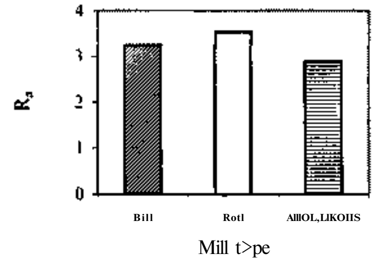
Figuie 4 Panicle size distiihution of calcite mineuil ground by (a) ball mill (h) lodımll te) autogtneous mill

Table I Suitace ıoughness values ot calcite mineial calculated from BET nıtıogen adsorption technique and measuied by a Suıtronıc *V* ınstıument

Mill type	ABET (mVgi)	RBET	R, (M»')
Ball	0 27	17 98	t 24
Rod	010	19 98	154
Aulogeneous	0 24	15 98	2 90



Figuie S The BET suitace roughness (R_BFT) values ot Iht pioducts of calcite giound in ditleient mills



l-iguie 6 The Suilionic V sulfate roughness (R) values of the puxluUs ot talute giouiul in different mills

The surlace roughness (RBCI) values determined by BET nitrogen adsorption technique weie correlated to the surface roughness values (RJ determined by Surtronic 3^+ technique ol calcite (Table 2) The established coirelation is shown in Figure 7 tor calcite mineial studied based on ball, rod and autogenous mills ıesults It is obviously seen that (RBLT) increases as the R, ıncıeases This means that it the suitace area ot calcite mineial measured by BET nitrogen adsorption technique is known, the surface roughness ot this mineral can be easily estimated or vice-versa

4 CONCLUSIONS

The surface roughness values obtained trom the BET surface aiea measurements were in the ıange ot 15 98-19 98 tor calcite mineial ground by ball, rod and autogenous mills The lower surface roughness (Rutr) value was 15 98 lor autogenous milled product, while the highest value obtained was 19 98 toi tod-milled calcite the surface roughness values, on the othei hand, obtained by Surtronic 3^+ were in the ıange ot 2 90-3 54 urn tor calcite mineral ground by ball, lod and autogenous mills The lower surface roughness (R,,) value was 2.90 um for autogenous milled calcite. while the highest value obtained was 3.54 urn for rod-milled product of calcite. In both techniques, the determined surface roughness values show good agreement and lower surface roughness values were obtained by autogenous milling comparing lo steel media charged mills. This is due to the abrasion mechanism that is in effect mostly in autogenous mills.

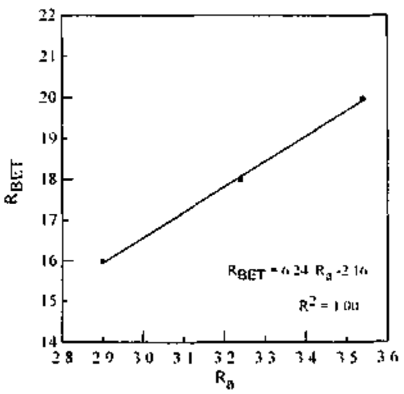


Figure 7. The variation of the RBET and R„ values

It was concluded that autogenous grinding generated particles having lower surface roughness than the other mills employed, i.e. ball and rod mills.

Therefore, the roughness values (RBET) determined by BET techniques of calcite particles increases (Figure 7) as the surface roughness values (R,,) determined by a Surtronic 3^+ increases. The empirical relationship found experimentally with a linear relationship is given as RBET= 6.24 R, - 2.16.

There is a correlation found between the RBET values and R, values for calcite mineral studied regardless of mill type used. As the RBET values increase, the R, values increase as well in the form of RBET= a. R, + b type of relationship, where a and b are constants (Figure 7). The R: value of this linear relationship found to be 1.00.

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NOMENCLATURE

ABET '• surface area measured by BET, enr/g
cl : density of solid, g/cnr[1]
D : particle diameter, cm
RBET '• surface roughness values determined by BET
R„ : average roughness, urn

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