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Dry grinding kinetics of binary mixtures of ceramic raw materials by Bond milling

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Abstract

The kinetics of batch dry grinding of binary mixtures of ceramic raw materials, namely quartz–kaolin, quartz–potassium feldspar and kaolin–potassium feldspar, from the feed sizes of −3.350 + 2.360, −2.000 + 1.400, −0.850 + 0.600, −0.500 + 0.355 and −0.300 + 0.212 mm have been determined using a Bond mill with a mixture of ball sizes of 38.10, 31.75, 25.40, 19.05 and 12.70 mm diameter and total mass of 22.648 kg. The Bond mill used was a size of 30.5 cm diameter, 30.5 cm length, with a total volume of 22,272 cm^3. The fractional ball filling was 22% of mill volume and the mill speed was 70 rpm. The breakage parameters were obtained for those mineral mixtures to predict the product size distributions. As the feed sized given above, which were ground in the mill, increase, the specific rate of breakage (S_i) values also increase, which means faster breakage with higher S_i value occurs in the order of quartz–kaolin, quartz–potassium feldspar and kaolin–potassium feldspar mixtures when comparing the characteristic α values (slope of S_i versus size relationship with higher α value). The cumulative breakage distribution function ($B_{i,j}$) values obtained for these mineral mixtures were slightly different in terms of the fineness factor, γ. This means that quartz–potassium feldspar mixture produced less fines with higher γ value, while kaolin–potassium feldspar gave more fines with lower γ value. The simulations of the product size distribution for these mixing were very close to the experimental data. Finally, slowing down effect, treated with false time concept, started earlier than the expected for these binary mixtures. There were some correlations found between the simulated time (θ) and experimental time (t).

Keywords: Binary mixtures; Breakage parameters; Bond milling; Kaolin; K-feldspar; Quartz

1. Introduction

Most of the energy is consumed in the grinding operations for ceramic industry. As it is known, some of the energy during grinding is converted to heat that is not utilized fully in grinding process. Thus, grinding is not very efficient operation and it needs to be taken care of in detail. However, it is possible to set a grinding system with low energy consumption and higher efficiency [1].

Ball milling has been used extensively in ceramic and mineral industries for more than a century. However, the fundamental mechanism of grinding is not understood fully yet. The early comminution theories were based on the empirical relationships between the energy input and reduction ratios. Since these approaches underestimate the breakage kinetics and some other sub processes in ball milling, they are caused some scale up problems in the industry. This issue made the grinding operation even worse which was not already an efficient process. Therefore, in order to remove these problems and make the grinding more efficient, kinetics of breakage approach in the mills has been developed as applicable models [2].

The description of grinding is split into two parts, selection and breakage. The selection function is defined as the fraction by weight of particles of a given size i which are selected and broken per unit time of grinding. The value

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Nomenclature

a_T	model parameter
$b_{i,j}$	breakage distribution function
$B_{i,j}$	cum. breakage distribution function
Q_i	correction factor
S_1	specific rate of breakage of feed size 1
t	grinding time
w_1	mass fraction of total charge of mill
W	total charge of mill
x_i	upper limits of the size

Greek symbols

α	model parameter
β	model parameter
γ	model parameter
Λ	model parameter
Φ	model parameter
θ	false time
μ	particle size at which correction factor is 0.5

varies with size and is denoted by S_i. The distribution function, $B_{i,j}$, is defined as the fraction by weight of breakage products from size j which fall below size i, where $i \leq j$ [3].

This paper reports on the kinetics of dry grinding of the binary mixtures of ceramic raw minerals when ground in a standard Bond mill, and presents the experimental results by comparing to simulated data.

2. Background

In the analysis of the breakage materials, it is useful to make the initial assumption that the breakage of each size fraction is first order in nature. That is, the rate of disappearance of size 1 due to breakage is proportional to the amount of size 1 material in the mill hold up [4].

$$-\frac{\mathrm{d}[w_1(t)W]}{\mathrm{d}t} \propto w_1(t)W \tag{1}$$

Since the mill hold up, W, is constant, this becomes:

$$\mathrm{d}w_i\frac{(t)}{\mathrm{d}t} = -S_1 w_1(t) \tag{2}$$

where S_1 is proportionality constant and it is called the specific rate of breakage, with units of time^{-1}. If S_1 does not vary with time [2]

$$w_1(t) = w_1(0)\exp(-S_1 t) \tag{3}$$

that is,

$$\log[w_1(t)] = \log[w_1(0)] - \frac{S_1 t}{2.3} \tag{4}$$

where $w_1(t)$ is the weight fraction of mill hold up that is of size 1 at time t [5]. The formula proposed by Austin et al [2] for the variation of the specific rate of breakage S_i with particle size is

$$S_i = a_T\left(\frac{x_i}{x_0}\right)^{\alpha} Q_i \tag{5}$$

where x_i is the upper limits of the size interval indexed by i, x_0 is 1 mm, a_T and α are model parameters that depend on the properties of the material and the grinding conditions. Q_i is a correction factor which is 1 for smaller sizes (normal breakage) and less than 1 (abnormal breakage) for particles too large to be nipped and fractured properly by the ball size in the mill. In abnormal breakage region, each size behaves as if it has some fraction of weak material and the remaining fraction of stronger material. Using a mean value for S_i in this region, values of Q_i are empirically described by

$$Q_i = \frac{1}{1 + (x_i/\mu)^{\Lambda}}, \quad \Lambda \geq 0 \tag{6}$$

where μ is the particle size at which correction factor is 0.5 and Λ a positive number which an index of how rapidly the rates of breakage fall as size increases that is the higher the value of Λ, the more rapidly the values decrease.

The cumulative breakage distribution, $B_{i,j}$, is defined as "The weight fraction of material broken from size j which falls less than the upper size of size interval i" is commonly used to characterize the size distribution resulting from breakage of material from a particular size interval to a smaller size [4,8].

The $B_{i,j}$ values can be calculated using the BII method which is described by [7].

$$B_{i,j} = \Phi_j\left(\frac{x_{i-1}}{x_j}\right)^{\gamma} + (1-\Phi_j)\left(\frac{x_{i-1}}{x_j}\right)^{\beta} \tag{7}$$

The parameter Φ is the intercept at $(x_{i-1}/x_j) = 1$, γ is the slope of the lower section of the $B_{i,j}$ curve and β is the slope of the steeper section of the $B_{i,j}$ curve as in Fig. 1 [2].

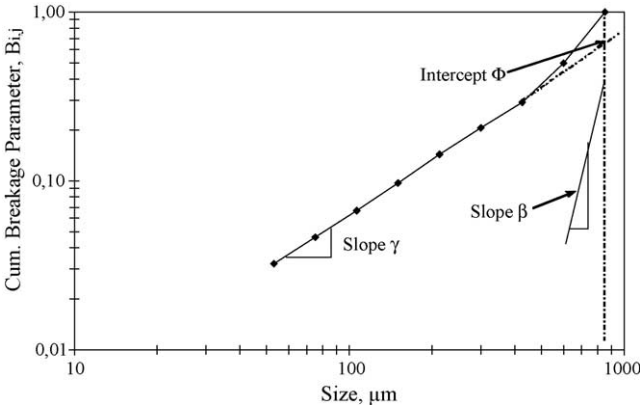

Fig. 1. Obtaining the primary breakage distribution function parameters for any single size fraction feed ground in the mill.

Table 1
Chemical composition of materials

	SiO_2	Al_2O_3	Fe_2O_3	TiO_2	CaO	MgO	Na_2O	K_2O	SO_3	LoI	Total
Quartz	99.62	0.20	0.04	0.01	0.04	0.03	0.01	0.02	0.00	0.04	100.00
Kaolin	59.93	27.86	0.33	0.82	0.06	0.00	0.03	0.00	0.50	10.47	100.00
K-feldspar	66.52	17.96	0.08	0.01	0.11	0.02	2.56	12.49	0.00	0.26	100.00

3. Materials and experimental methods

3.1. Materials

The binary mixtures (1:1) of quartz, kaolin and potassium feldspar were chosen as the feed minerals for this study, because these minerals are major raw materials of ceramic industries. The densities of these raw materials, measured by a pycnometer, are averaged as 2.68, 2.61 and 2.63 g/cm^3 over thirteen measurements and Mohr's hardness of the same three minerals, measured by a hardness pen, are 7, 3, 6, also work indexes (W_i) of these materials are 11.38, 10.52, 11.14 kWh/t, respectively. The work indexes of quartz–kaolin, quartz–potassium feldspar and kaolin–potassium feldspar mixtures are 10.95, 11.26, 10.83 kWh/t, respectively. The Bond work index is determined by using the standard Bond test procedure [2]. The experimental procedure to determine the work indexes of minerals are also given in Appendix A. Chemical analyses of these materials are also given in Table 1.

The breakage parameters were determined experimentally using one size fraction technique [8]. Tests were carried out on 1:1 binary mixtures of these raw materials. The size fractions chosen for tests were −3350 + 2360, −2000 + 1400, −850 + 600, −500 + 355 and −300 + 212 μm, where for example −3350 + 2360 μm denotes that 100% of the particles are passing by weight at 3350 μm size and 100% of particles are remaining at 2360 μm.

3.2. Experimental methods

The stainless steel Bond mill used in the experiments was 30.5 cm in diameter and 30.5 cm in length. The mill was loaded with 22.648 kg of 38.1, 31.75, 25.40, 19.05 and 12.70 mm diameters of stainless steel balls which correspond to 22% of the struck volume of the mill and mill was operated at 70 rpm which was 86% of its critical speed. In each case, the mill was loaded such that 100% of the interstitial void volume of the ball charge was filled with the mono-sized material at the beginning of the experiments for the minerals mixed in 1:1 ratios by volume. The Bond mill developed by F.C. Bond is universally used for grindability testing, including determination of the Bond Work Index (W_i). A charge of 285 iron balls ranging from 15.2 to 44.4 mm diameter and weighting 20.648 g is used. The characteristics of the Bond mill used in grinding tests and test conditions are outlined in Table 2.

In order to obtain the breakage parameters of each binary mixture, the mixtures were ground dry for 0.5, 1.0, 2.0, 4.0,

Table 2
Bond mill characteristic and test conditions for grinding of ceramic raw materials

Mill	Diameter, D (cm)	30.5				
	Length, L (cm)	30.5				
	Volume, V (cm^3)	22,272				
	Speed (rpm)	70				
	Critical speed, N_c[a]	86.55				
Mill charge	Diameter, d (mm)	38.10	31.75	25.40	19.05	12.70
	Number	43	67	10	71	94
	Total mass (g)	22,648				
	Specific gravity (g/cm^3)	7.79				
	Fractional ball filling, J[b]	0.22				
Media charge	Sample	Quartz		Kaolin	K-feldspar	
	Specific gravity (g/cm^3)	2.68		2.61	2.63	
	Powder weight (g)	1,555.2		1,514.6	1,526.2	
	Fractional. Powder filling, f_c[c]			0.08		
	Powder-ball loading ratio, U[d]			1.00		

[a] $N_c = \frac{42.3}{\sqrt{D-d}}$.

[b] $J = \left(\frac{\text{mass of balls/ball density}}{\text{mill volume}}\right) \times \frac{1.0}{0.6}$.

[c] $f_c = \left(\frac{\text{mass of powder/powder density}}{\text{mill volume}}\right) \times \frac{1.0}{0.6}$.

[d] $U = \frac{f_c}{0.4J}$.

6.0, 9.0 min. At the end of the each run, the ground product was weighed and the loss was no more than 0.15% of the total charge. Due to grinding, sampling, sieving and regrinding the same sample for next time, some weight losses are expected. The loss in our study was also in acceptable range, which is less than 1%.

Mono-sized quartz–kaolin, quartz–K-feldspar and kaolin–K-feldspar mixtures were prepared to give 1:1 mixtures on a volume basis would fill ball void in the mill. The amounts of quartz, kaolin and K-feldspar charged into the mill for grinding were 1.555, 1.514 and 1.526 kg, respectively. These amounts were calculated and also given in Table 2 as ''mass of powder''.

The size fractions in the ground products were analyzed for their quartz, kaolin and K-feldspar contents by inductively coupled plasma (ICP) technique. Elemental concentrations can be measured to ppb (parts per billion) levels using the inductively coupled plasma emission spectrophotometer. The ICP spectrometer utilizes plasma to excite elemental electrons that produce photons unique to each element. An example of procedure is one used for a whole rock analysis. In this procedure a lithium metaborate flux is generally used to digest the specimen. Once digested, the solution is introduced to the plasma allowing elemental concentration comparisions to know concentration curves. Using stoichiometric techniques elemental concentrations can be converted into moleculer weight percentages. This method atomizes and excites even the most refractory elements with high efficiency. With this ICP, several elements can be determined simultaneously without the need for repeated aspirations, adjustment of instrument parameters and tracking of the samples.

3.3. Simulation

The simulation of the product size distributions of the grinding tests results for quartz, kaolin and potassium feldspar mixtures were done by using the simulation program called PSUSIM [9,10].

The simulator consists of a ball mill model (with a number of options for the residence time distribution) plus linking algebra to enable two mills to be connected in any desired circuit. The program is written in IBM Basic, and is constructed in modular form, to allow easy modification of mill models, classifier models, etc. The simulation program needs the input of characteristic breakage parameters for the mineral of interest ant that these are often determined in a small-size laboratory ball mill and scaled up by the program to the conditions of full-scale ball mill [9,10].

The simulation of the product size distributions were made by selecting the batch-open circuit option in the simulator, then entering the S_i and $B_{i,j}$ parameters obtained from the laboratory experiments as the characteristics breakage parameters.

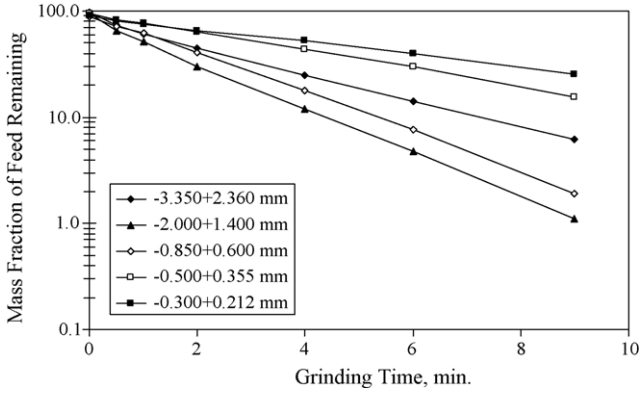

Fig. 2. First order plots for dry grinding of different feed sizes of quartz–kaolin binary mixtures.

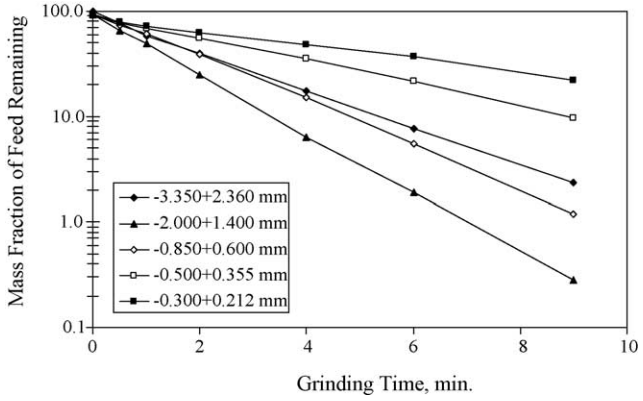

Fig. 3. First order plots for dry grinding of different feed sizes of quartz–K-feldspar binary mixtures.

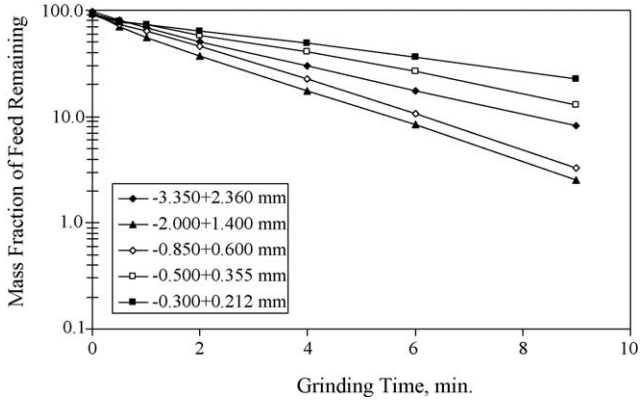

Fig. 4. First order plots for dry grinding of different feed sizes of kaolin–K-feldspar binary mixtures.

4. Results and discussion

4.1. Breakage rate functions

Figs. 2–4 show the first order plots for the binary mixtures of minerals for various size fractions when ground as 1:1 binary mixtures. The results indicated that breakages

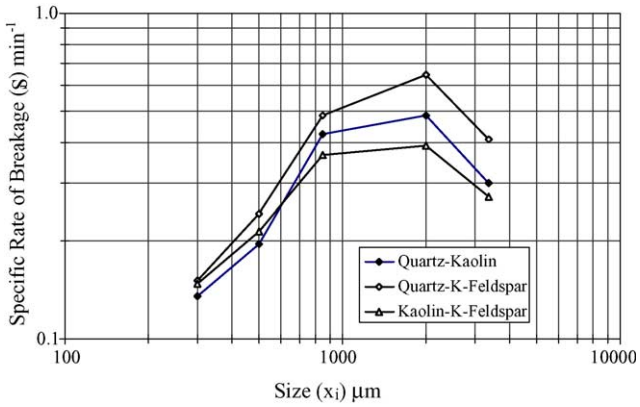

Fig. 5. Variation of S_i values of binary mixtures of quartz, kaolin and K-feldspar with particle size.

generally followed the first order disappearance kinetics and values of S_i could be determined easily.

Fig. 5 also shows the variations of the specific rates of breakage, S_i, values with the particle feed sizes ground in the mill for the minerals mixed in 1:1 ratios. It is clearly seen that S_i values increase up to a maximum particle size (2000 μm), then start decreasing at around 2000 μm for all materials in Fig. 5. This was due to the in efficiency of the largest feed sizes that were not nipped properly by the balls in the mill. This means the feed sizes larger than 2000 μm will not be ground efficiently in this mill with these ball sizes (Fig. 6).

The initial grinding results obey the first-order breakage form as in Eq. (5). When the values of S_i are fitted to the expression (5); the a_T value is obtained by inserting α, x_i and x_0. Using Eq. (5), S_i, a_T, α, μ and Λ were obtained for all mineral mixtures and outlined in Table 3 as the breakage parameters to use for simulations of the product size distributions. The S_i values were obtained from the first order plots (see Figs. 2–4). Using S_i values obtained from these figures versus particle feed size (upper size of the fraction) is plotted as in Fig. 5. The slope of the curve is α, the S_i value at 1000 μm (x_0) gives the a_T value. The μ and Λ parameters were calculated using Eqs. (5) and (6).

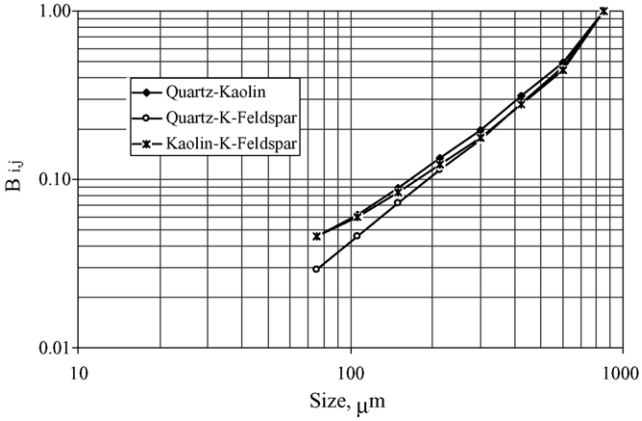

Fig. 6. Primary breakage distribution functions of binary mixtures of quartz, kaolin and K-feldspar.

Table 3
Characteristic breakage parameters obtained from the laboratory test to use in the simulator

	a_T	α	μ	Λ	Φ	α	β
Quartz–kaolin[a]	0.64	1.45	1.44	2.88	0.50	1.00	4.70
Quartz–K-feldspar[b]	0.67	1.32	1.71	2.90	0.55	1.40	7.71
Kaolin–K-feldspar[c]	0.54	1.13	1.49	2.39	0.49	0.94	4.64

[a] 50% quartz, 50% kaolin mixture.
[b] 50% quartz, 50% K-feldspar mixture.
[c] 50% kaolin, 50% K-feldspar mixture.

4.2. Breakage distribution functions

From the size distributions at the shortest grinding times, the values of cumulative breakage distribution functions, $B_{i,j}$, which is commonly used to characterize the size distributions resulting from breakage of material from a particular size interval to a smaller size were determined using the BII method [2,5,6]. The values of $B_{i,j}$ against particle size obtained from BII calculations for the −850 + 600 μm size fractions are plotted in Fig. 6. In order to get the $B_{i,j}$ values, BII calculation procedure [2] given below was applied for the shortest grinding time (0.5 min),

$$B_{i,j} = \frac{\log[(1 - P_i(0))/(1 - P_i(t))]}{\log[(1 - P_2(0))/(1 - P_2(t))]}, \quad i > 1 \tag{8}$$

where $P_i(0)$ = cumulative weight fraction of time 0 for ith interval, $P_2(0)$ = cumulative weight fraction of time 0 for second interval, $P_i(t)$ = cumulative weight fraction of time t for interval t, $P_2(t)$ = cumulative weight fraction of time t for second interval.

The $B_{i,j}$ values of the binary mixtures determined Eq. (7) are also given in Table 3. As it is easily seen, the $B_{i,j}$ values of the −850 + 600 μm feed were obtained to be slightly different from each other. The $B_{i,j}$ values of the other feed size fractions studied were obtained to be very close to each other as well. That's why we presented −850 + 600 μm size fraction data in this study. The approach used for the −850 + 600 μm size fraction to get the breakage parameters is the same as the other size fractions.

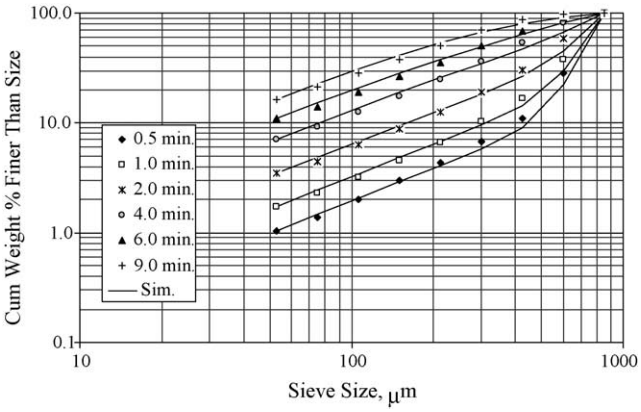

Fig. 7. Simulated and experimental product size distribution of quartz–kaolin mixtures for −850 + 600 μm feed fraction.

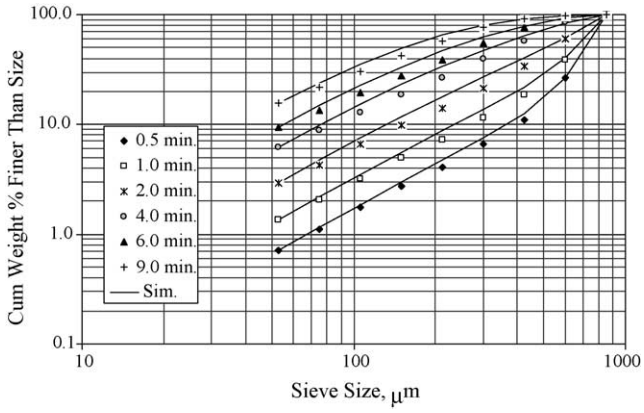

Fig. 8. Simulated and experimental product size distribution of quartz–K-feldspar mixtures for −850 + 600 μm feed fraction.

4.3. The product size distribution and simulation of binary mixtures of quartz, kaolin and potassium feldspar

Figs. 7–9 show the experimental and simulated particle size distributions of binary mixtures of quartz, kaolin and potassium feldspar for the feed size fraction of −850 + 600 μm only. The simulations gave good agreement with the experimental data, i.e., the simulated data points are very close to the experimental data points. The differences between those data points are no more than 1%, which is good. However, the slowing down effect in the mill started at the beginning of the grinding times for each binary mixture, particularly kaolin–K-feldspar mixture that showed this effect at 0.32 min. Here, the slowing down effect was treated using the false time or calculated time concept by making the simulation program produce a match to a specified point on the product size distributions and designation the grinding time (t) necessary to achieve this match as the calculated time, θ, where $\theta \leq t$ as given in Fig. 10 [11,12].

Fig. 10 thus shows these relationships between the calculated time and the real time for all mixtures ground. The empirical equations were also obtained with "$\theta = a \times t + b$"

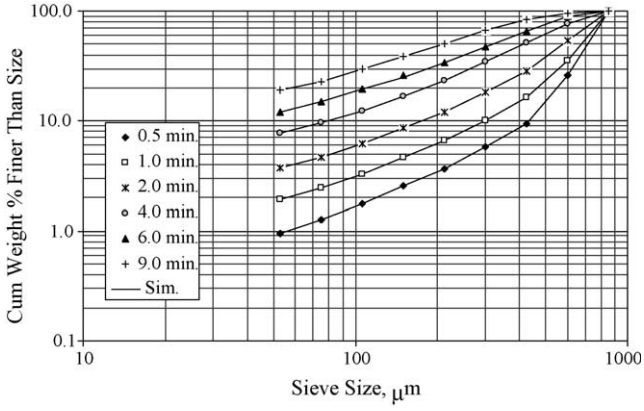

Fig. 9. Simulated and experimental product size distribution of kaolin–K-feldspar mixtures for −850 + 600 μm feed fraction.

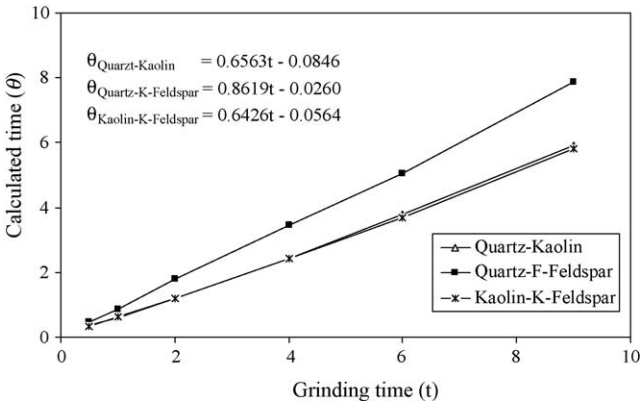

Fig. 10. False time (θ) versus real grinding time (t) for binary mixtures studied.

type of relations to set the actual grinding time for the mill to obtain the desired size distributions.

5. Conclusions

(1) Faster breakage (shown by α parameters in Table 3) from higher value to lower ones were in the order of quartz–kaolin, quartz–K-feldspar and kaolin–K-feldspar mixtures for the feeds of −850 + 600 μm fractions. The α values were obtained to be 1.45, 1.32 and 1.13 for the above order, respectively. All other feed sizes studied gave same tendency in terms of the specific rates of breakage values.

(2) The $B_{i,j}$ values (Φ, β, γ) were obtained to be slightly different for each mixtures; the characterization parameter (γ), which is fineness factor was the highest for quartz–K-feldspar binary mixture indicating less fines produced below ∼200 μm, while the lowest γ value was obtained for kaolin–K-feldspar mixture which produced more fines in the mill (broader size distribution in the fines region: i.e, below ∼200 μm). Quartz–kaolin binary mixture remained in between of the other two mixtures according to the γ values.

(3) The slowing down effect in the Bond mill started earlier then expected. This is probably due to the difference of the Bond mill that does not have any lifters and its dimension comparing to a standard ball mill design. Moreover, the mixtures of minerals ground were not brittle with the exception of quartz and they were not ground alone to show their own grinding characteristics. Here they were affected from each other when ground in mixed portions.

(4) The simulations of the product size distributions for the all mixtures were in good agreement with the experimental data.

(5) The slowing down effect, treated with the calculated or false time (θ) in connection to real grinding or experimental grinding time (t), was accomplished with "$\theta = a \times t \pm b$" type of linear equation (see Fig. 10).

The equations found were $\theta = 0.66t - 0.08$ for quartz–kaolin mixture, $\theta = 0.86t - 0.03$ for quartz–K-feldspar mixture and finally $\theta = 0.64t - 0.06$ for kaolin–K-feldspar mixture. The use of these equations provides us to predict the operating grinding time to set for the desired product size distributions of any mineral mixtures studied.

(6) The breakage parameters obtained from the laboratory studies are applied to industrial applications by choosing the scale-up option in the program for the mixtures of any minerals.

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Appendix A

Test results were converted to a work index by the following empirical equation:

$$W_i = \frac{44.5}{P_t^{0.23} G_e^{0.82} [10/P_{80}^{0.5} - 10/F_{80}^{0.5}]} (\text{kWh/st})$$

where P_t is the selected test size, in microns; G_e is the grindability t steady state conditions, in g/rev; P_{80} is the 80% passing product particle size, in microns; F_{80} is the 80% passing feed particle size, in microns.

The value of W_i obtained by above equation is representative of the required energy to tumble the charge of a 30.5 cm interior diameter overflow ball mill, grinding wet and in closed circuit.

Estimation of W_i is realized only after attainment of steady-state conditions for a locked-cycle batch-grinding test. A grinding cycle consists of the following two parts:

1. Dry grinding of 700 cm^3 bulk volume of material prepared to −3350 microns in a 30.5 cm × 30.5 cm ball mill rotating at 70 rpm and charged with 22.648 gm of grinding balls.
2. Removal of sieving of the portion of the product, which was ground finer than a pre-selected test size. In preparation for the next cycle, the undersize portion is replaced by n equivalent amount of fresh feed in order to maintain constant charge.

The duration of grinding is established as the time required to produce, for the next cycle, a simulated 250% re-circulating load of particles coarser than the selected test size. An estimate of the grinding time is obtained using the grindability, G, of the previous cycle. G is defined as the grams of particles finer than test size produced per mill revolution. An initial value of G is obtained by waiving the 250% re-circulating load requirement for the first cycle and replacing it with a grind period of 100 million revolutions. When G reverses its direction and remains constant from cycle to cycle, steady-state grinding conditions are said to prevail. The average value of G for the final three cycles is taken as G_e, the steady-state grindability.

Selection of the test size, P_t, is usually based on the fineness of grind desired in the milling operation. Typical choices for P_t are 106, 75 and 53 μm. Care must be taken to minimize the possibility of sieve blinding. Blinding will cause underestimation of G, with direct consequences on the calculate value of W_i.

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