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The effect of microwave radiation on grinding kinetics by selection function and breakage function - A case study of low-grade siliceous manganese ores

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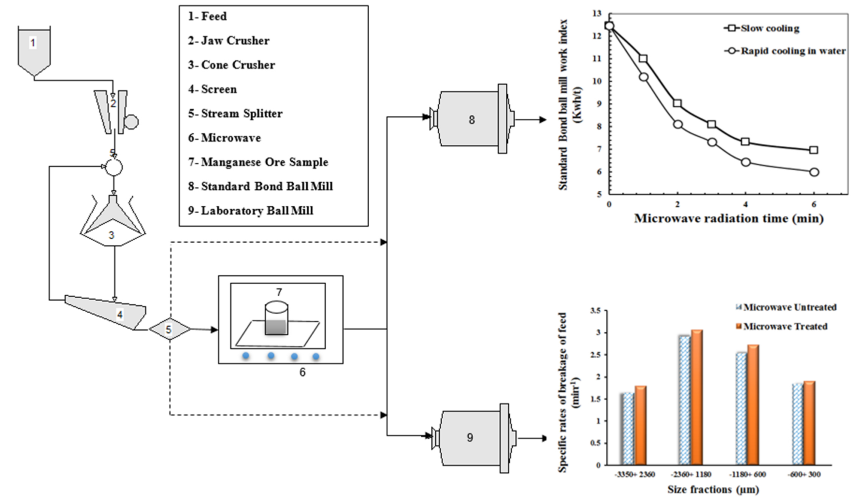
HIGHLIGHTS

- Improvement of grindability ore by microwave treatment.
- Intergranular cracks formed between hematite with gangues minerals after microwave treatment.
- Increasing the special rate of breakage of manganese ore up to 8.42% using microwave treatment.
- The microwave treated products are coarser than the microwave untreated products.

GRAPHICAL ABSTRACT



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ABSTRACT

In this study, the effect of microwave radiation on grindability and grinding kinetics were investigated. Microwave treatment was performed using an oven with 1100 W power and 2.45 GHz frequency. In order to study the breakage mechanism the grindability from the standard Bond ball mill work index (BBMWI) test was used with the selection function and breakage function as grinding parameters for treated and untreated samples. Based on the results of grindability, the work index (W_i) of a standard Bond ball mill after 4 min of microwave radiation decreased from 12.46 kWh/t to 6.45 kWh/t. selection function results showed that the specific rate of breakage (S_i) value for the size fraction −3350+2360 µm increased to 8.42% after microwave treatment. Cumulative breakage function results showed that microwave-treated products were coarser in comparison with untreated products. This phenomenon is more significant in coarse fractions, where the effect of microwave treatment is more obvious.

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1. Introduction

Microwave is a non-ionizing electromagnetic radiation with frequencies in the ranges of 300 MHz to 300 GHz and the microwave wavelength is from 1 to 300 mm which can penetrate deeply into the sample. The most commonly utilized frequency for a home microwave oven is 2450 MHz [1,2]. Materials may be classified into three groups, i.e. conductors, insulators, and absorbers. Materials that absorb microwave radiation are called dielectrics. The interaction of dielectric materials with electromagnetic radiation in the microwave range results in energy absorbance. As all materials cannot be heated rapidly by microwaves, the differential heating rates occurring in the minerals of the material can induce internal thermal stress [3,4].

Microwave treatment has been identified as a pre-treatment method which has attracted the attention of researchers of mineral processing in recent years. A number of microwave application processes have been investigated. These include microwave assisted liberation, microwave assisted ore grinding [5,6], electrostatic separation [7], magnetic separation [8, 9], flotation [10] and microwave assisted minerals leaching [11]. One of the most important potential applications of microwave treatment is optimizing ore grinding to reduce operating cost. Kingman *et al.* [12] investigated microwave treatment of copper carbonatite ore using a single mode. The results showed that microwave treatment led to a reduction of up to 30% in grinding energy. QEMSCAN analysis of the product drop weight tests also showed a decrease in the amount of locked and middling copper sulfide in the +500 µm size fraction. Sikong *et al.* [13], carried out an investigation on the thermal treatment of granite rock. The results showed that the strength of treated granite is less than 60% of the original after 30 minutes of exposure. Also, Song *et al.* [14] investigated the effect of microwave treatment on Oolitic iron ore. Microwave radiation led to micro cracks in oolitic iron ores along the boundaries of the hematite and gangue minerals. It was shown that at the same size fractions the microwave treatment increased the liberation of hematite and gangue minerals about 20-30%.

Despite the studies performed on microwave treatment, few studies have investigated the effect of microwave treatment on breakage and selection functions. The effect of microwave treatment on the specific rate of breakage of coal ore was investigated by Sahoo *et al.* [15]. The results showed that microwave treatment led to a 15% increase in the specific rate of breakage. Also, Koleini *et al.* [16] studied the effect of microwave treatment on grinding kinetics of iron ore. The results showed that the specific rate of breakage (S_i) increased by an average of 50%. In addition, it was found that microwave-treated iron ore produced coarser material than untreated iron ore considering the γ value of $B_{i,j}$.

The aim of this study is to investigate the effect of microwave treatment on grindability of siliceous manganese ores with the standard Bond ball mill work index. To study the breakage mechanism, selection and breakage function were used as grinding parameters at treated and untreated conditions.

2. Experimental methods

2.1. Materials and equipments

The sample used in this study was obtained from the Venarj Manganese ore mine located 27 km south west of Qom Province, central Iran. An X-ray diffraction analysis (XRD) model PW1800 with a copper tube and polarized optical Zeiss Axioplan 2 microscope with light reflection and transmission were used to investigate the minerals present in the sample and identify their type and relative abundance, respectively. A chemical analysis was performed on all sample powders by X-ray fluorescence (Fillips PW 1480 model).

2.2. Software calculation

Excel Software was used to calculate the breakage and selection functions of the experiment. Data fitting was done by minimizing the sum of error squares with Excel.

2.3. Methods

2.3.1. The grindability test

To determine the grindability from a standard Bond ball mill (Labtech Essa Pty Ltd, Australia), with 305 mm diameter and 305 mm length, critical speed runs at 70 rpm were used. The grinding charge includes 285 steel balls and has a total mass of 21.824 kg. Samples with 700 ml volume (1400 g) were used in order to determination the standard Bond ball mill work index, from a feed of –3.35 mm. Figure1 shows the size distribution of the feed sample of Bond ball mill that was used in the study. F80 of the feed sample was obtained by sieve analysis, which is equal to 2288 μm. The work index tests on microwave treated samples were carried out at different times. Eq. (1) was used to calculate the standard Bond ball mill work index [17].

$$W_i = \frac{45.39}{P_i^{0.23} G_i^{0.82}} \times \left[\frac{1}{\frac{10}{\sqrt{P_{80}}} - \frac{10}{\sqrt{F_{80}}}}\right] \quad (1)$$

where, W_i: work index (Kwh/t); F_{80}: 80% passing size of original feed (μm); P_{80}: 80% passing size of circuit product (μm); G_i: produced fine particles in a cycle (g/rev), and P_i: the test-sieve size (μm).

2.3.2. The grinding kinetics

The selection and breakage functions were used in order to investigate the grinding mechanism. The strength of the microwave used for this experiment was 1100 W and its frequency is 2.45 GHz. In addition, four Single-sized fractions of siliceous manganese ores (−3350+ 2360, −1700 + 1180, −1180+ 600 and −600+ 300 μm) were prepared. Two samples from each fraction weighting 300 g were prepared. Grinding tests were performed in a laboratory ball mill of 20 cm diameter and 25 cm length and critical speed of 76 rpm (80% of the critical speed). The mill charge consisted of stainless steel balls ranging from 15–35 mm in diameter, the total weight of the grinding charge was 7700 g. To evaluate the effect of the microwave treatment, one sample was exposed to the cavity for 4 min at 1100 W and was then compared with the untreated sample. Each fraction of ore was ground for a short time period and then the mill content was discharged and sieved. The sample was poured into the mill again until about 40-50% of the material was passed through the top screen. After the grinding test, particle sizes of interest were split into number size intervals with aperture ratio of √2-spaced sieves to determine size distributions.

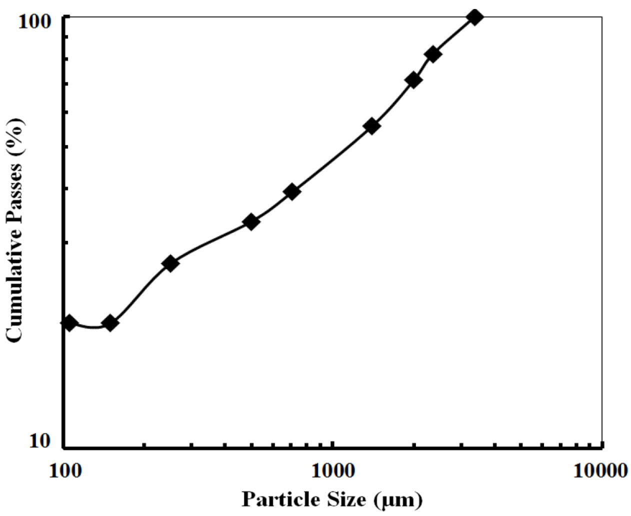

Fig. 1. Particles size distribution of manganese ore.

3. Results and discussion

3.1. Characterization of siliceous manganese samples

3.1.1. Mineralogy of the sample

Mineralogical studies revealed that the major minerals present in the sample were hematite, braunite, goethite, quartz, and calcite. Braunite is mainly interlocked with quartz, calcite and clay minerals (Figure 2a) of mainly triple types, which decreased with particle size (Figure 2b).

3.1.2. XRD analysis

The XRD analysis sample is shown in Figure 3. The results show that hematite (Fe_2O_3), braunite ($Mn^{2+}Mn_6^{3+}SiO_{12}$), quartz (SiO_2) and calcite ($CaCO_3$) are the main minerals in the sample and montmorillonite ($(Ca_{0.2}Al,Mg)_2Si_4O_{10}(OH)_2,xH_2O$), orthoclase ($KAlSiO_3O_8$), and albite ($Na,Ca(Si,Al)_4O_8$) are minor minerals. Montmorillonite is the only hydrated mineral in the sample.

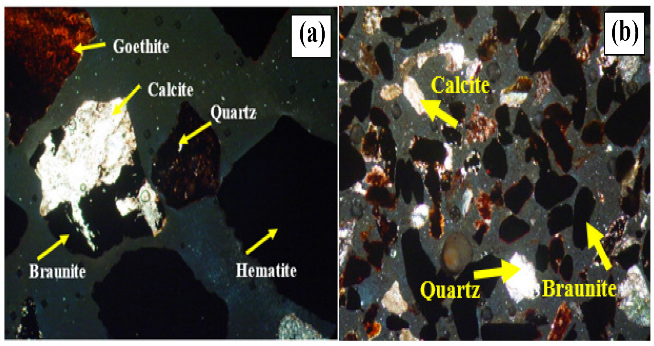

Fig. 2. Microscopic images. (a) Inter-locked braunite with calcite, quartz, clay mineral and (b) Particles liberated in size fraction -150 μm.

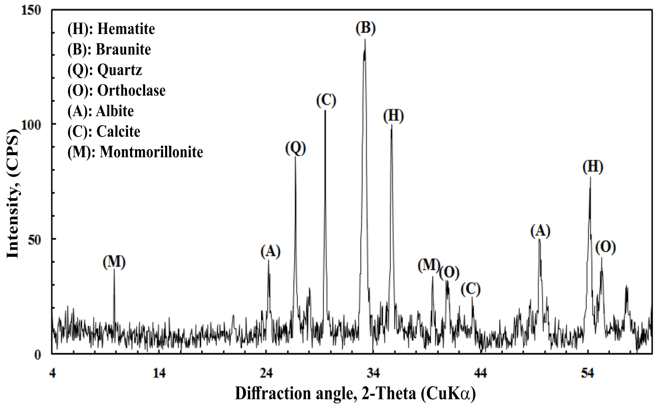

Fig. 3. XRD graph of the siliceous manganese sample.

3.1.3. XRF Analysis

Table 1 shows the chemical analysis of the manganese sample. The results of the analysis showed that the sample included: Fe_2O_3: 31.51%, MnO: 16.97%, SiO_2: 26.90 % and CaO: 8.31%.

3.1.4. Microwave heating effect on the sample

Based on the results of previous researchers, minerals behave differently in microwave radiation and are classified based on heating rate into hyperactive as active, difficult-to-heat and inactive [18]. Hematite, goethite and manganese minerals are active materials during microwave treatment while gangue minerals (quartz, calcite) are inactive [18,19]. When hematite, goethite and manganese minerals are exposed to microwave radiation, hematite, goethite and manganese minerals expands more than gangue minerals and the difference caused by this expansion results in the formation of inter-granular fractures. Table 2 shows the heating behavior of hematite, goethite and manganese minerals as well as gangue minerals.

Table 2. Heating properties of minerals with microwave radiation [18,19].

Mineral	Formula	Microwave heating
Hematite	Fe_2O_3	Heat readily
Goethite	FeO(OH)	Heat readily
Braunite	$Mn^{2+}Mn_6^{3+}SiO_{12}$	Heat readily
Calcite	$CaCO_3$	Does not heat
Quartz	SiO_2	Does not heat

3.2. The grindability test

The variation of standard Bond ball mill work index of the siliceous manganese ore at various times under microwave radiation and ore-cooling condition is shown in Figure 4. The results showed that the work index decreased with an increase in microwave radiation time. This may be due to the differences in dielectric properties in the mineral, which led to an increase in grindability (decreased in work index). According to Figure 4, quenching (rapid cooling in water) led to a greater decrease in the Bond ball mill work index. When the particles are rapid cool in water the outer layers contract more rapidly than the center. This creates tensile stresses which cause the particles to break and crack. As can be seen in the figure, an increase in time of microwave radiation to 4 min decreased the amount of work index from 12.46 kWh/t to 6.45 kWh/t (a 48% decrease).

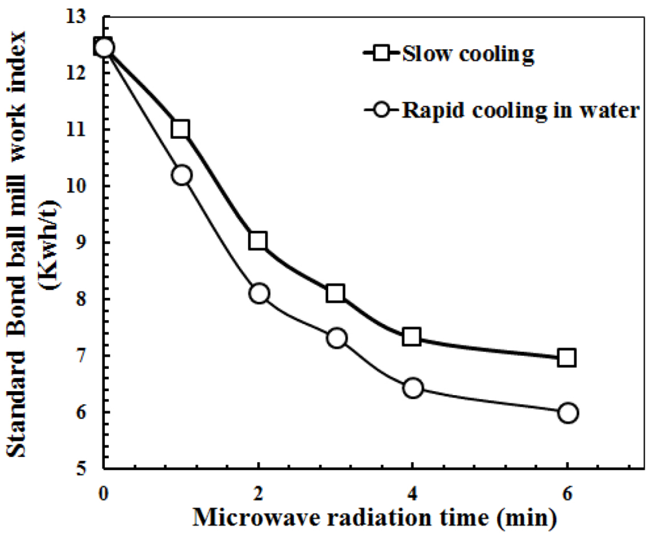

Fig. 4. Effect of microwave radiation time on standard Bond ball mill work index.

Table 1. Chemical analysis (XRF) of the siliceous manganese sample.

Composition	Fe_2O_3	SiO_2	MnO	CaO	Al_2O_3	K_2O	P_2O_5	As_2O_3	TiO_2	L.O.I
Mass (%)	31.51	26.90	16.97	8.31	7.06	1.18	0.13	0.26	0.53	7.15

3.3. Selection function

The rate of disappearance due to breakage in the largest size can be described by the following first-order equation [20]:

$$\frac{dm_i}{dt} = -S_i m_i \qquad (2)$$

where S_i (min^{-1}) is the rate of breakage out of size fraction i (selection function) and m_i is the mass fraction in size i after a grind time, t. When Eq. (2) is integrated it gives:

$$\log m_1(t) - \log m_1(0) = -S_1 t/2.303 \qquad (3)$$

Plotting experimental values of $m_1(t)$ versus t on log-linear scales, the slope of the line is equal to S_1.

3.3.1. The influnce of mass retained with grinding time

The influence of mass retanied versus grinding time for the −1700+1180 μm fraction of microwave-treated and untreated samples is showed in Figure 5. It can be observed that the microwave treated sample initially grinds much more rapidly than the untreated sample. Figures 6 and 7 show the plots of the selection function for different fractions of microwave-treated and untreated samples. According to Figure7, specific rates of breakage (S_i) in both conditions of treated and untreated samples are first-order kinetics, but S_i has obvious non-first-order kinetics for –3350+2360 μm (Figure 6). This may be caused by the existence of very coarse particles in comparison with the ball size used in the laboratory mill. In this size fraction, which cannot be crushed by balls or with passing of time, accumulation of coarse and hard particles on the first screen causes the deviance from first order kinetics.

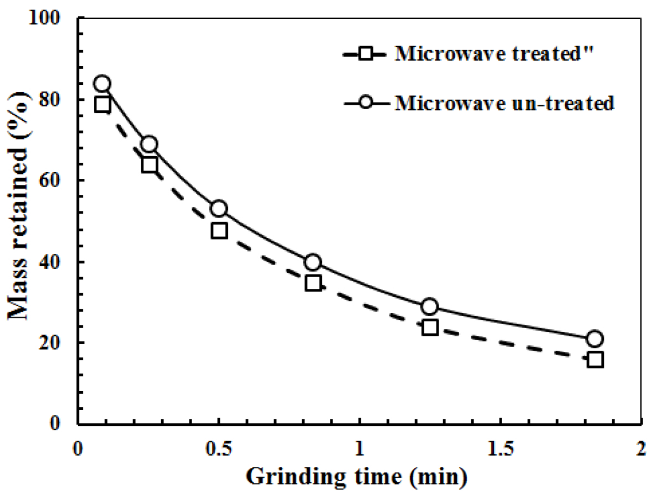

Fig. 5. The influence of mass retained with grinding time for -1700+1180 μm fraction.

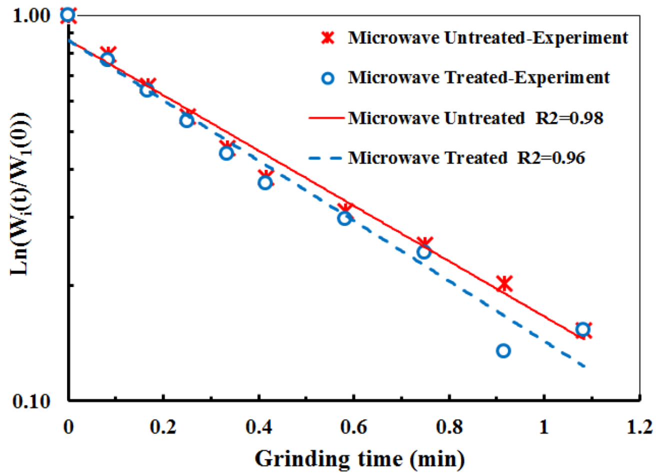

Fig. 6. The percentage oversized versus grinding time for batch dry grinding of feed -3350+2360 μm.

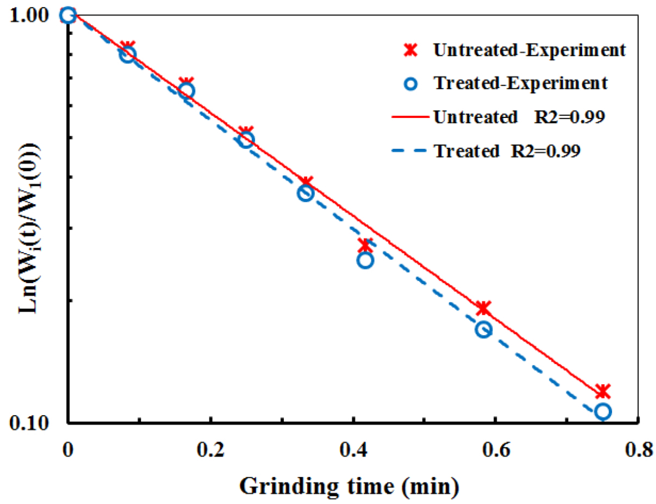

Fig. 7. The percentage oversized versus grinding time for batch dry grinding of feed -1700+1180 μm.

The Si values for various size fractions are shown in Figure 8. The S_i values are estimated from the average of the plots. The results show that after microwave treatment, the increase in S_i value occurs as particle size increases. It is obvious that at –3350+2360 μm fraction, about 8.42% increases in specific rates of breakage (from 1.65 to 1.79 min^{-1}) were achieved after microwave treatment.

3.4. Breakage function

When a material of size j breaks once, the mass

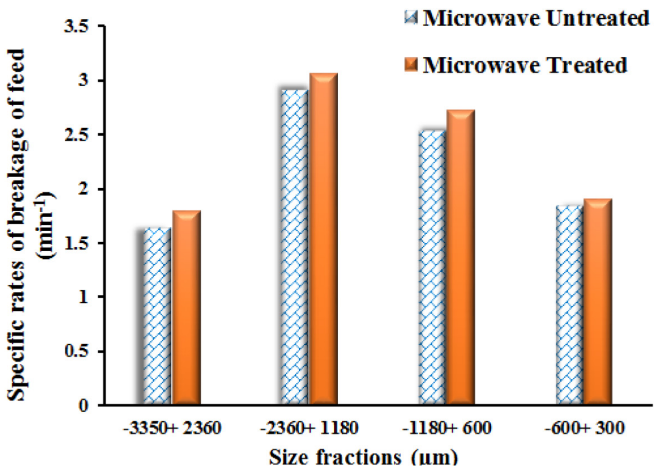

Fig. 8. Specific rates of breakage of feed against size fractions.

fraction of broken products with size i can be represented by the breakage function $B_{i,j}$, in which size i is smaller than size j. Furthermore, for short grinding times the values of $B_{i,j}$ can be estimated from a size analysis of the product, using a narrow particle size fraction j as an initial feed through the BII method [21,22].

$$B_{i,j} = \frac{\log[1 - P_i(0)/(1 - P_i(t)]}{\log[1 - P_{j+1}(0)/(1 - P_{j+1}(t)]} \qquad i > j \qquad (4)$$

where P_i (t) is the mass fraction less than size i at time t. Eq. (3) presupposes that a small mass fraction of particles will re-break into smaller sizes. Breakage function ($B_{i,j}$) for all size fractions is obtained from Eq. (3). The Broadbent and Callcott equation was used for data fitting and obtaining the model parameters, the cumulative breakage function $B_{i,j}$ can also be represented by the following empirical relation:

$$B_{i,j} = \varphi(\frac{X_i}{X_1})^{\gamma} + (1 - \varphi)(\frac{X_i}{X_1})^{\beta} \qquad i > j \qquad (5)$$

where X_i/X_1 is the relative particle size and parameters γ, β, and φ are the characteristics of the material being ground and define the size distribution [23,24]. β was kept constant for better data fitting. Data fitting was done by minimizing the sum of error squares with Excel.

The Breakage function values for −3350+2360 µm fraction in both the microwave treated and untreated samples are shown in Tables 3 and 4. According to the results obtained from the breakage function, breakage function values ($B_{i,1}$) of the microwave treated sample decreased. This means that the amount of materials passing through a specific screen is less than the untreated condition after the first break in microwave radiation.

Breakage parameters (φ,γ) in microwave-treated and untreated conditions are illustrated in Figures 9 and 10 (β is constant in all situations). One of the most important breakage parameters is γ which shows the breakage behavior of the sample. According to the previous researches [25], γ describes the amount of fine particles produced from the top screen. Higher amounts of γ mean that the size of particles resulting from breakage is close to the first screen, and this happens when the grinding rate is slow. In other words, a lower amount of γ would imply more effective breakage action with high production of fine particles. Overall, softer materials would display lower values of γ as compared with harder materials.

As mentioned in the Breakage Function, microwave-treated samples have coarse products after grinding; furthermore, information from Figure10 indicates increment in γ for microwave-treated samples in all size fractions and this effect is significant in coarse sizes such as size fraction of −3350+2360 µm which showed a 30% increase in γ from 0.5293 to 0.6904. Therefore, it can be said that microwave treatment of samples causes a treated sample with coarse particles to produce more particles close to the fractured sample. Other investigations that have studied the influence of microwave treatment on the breakage function have shown the same results. Koleini *et al.* [16], studied the effect of microwave treatment on iron ore. According to the obtained results, breakage functions ($B_{i,1}$) decreases during the microwave treatment. This reduction is more observable for coarser size fractions. Also, the effect of microwave treatment decreases as the size reduces because the liberation degree of minerals increases [26].

4. Conclusion

The investigation of microwave treatment influence on breakage and selection functions of ball mill showed that fine fractions of this materials follow the first-order kinetic, but the specific breakage rate of coarse fraction (−3350+2360 µm) deviates from the first-order kinetic. The specific rate of breakage (S_i) value for a size fraction of (−3350+2360 µm increased 8.42% after microwave treatment. Cumulative breakage function results showed that treated products are coarser in comparison with untreated products. This phenomenon

Table 3. The breakage function values for the microwave untreated -3350+2360 µm fraction.

Particle size (µm)	Relative particle size	Breakage function		Error	Squared error
		Experiment	Model		
2360	1	1	1	0	0.0000
1700	0.720	0.4841	0.4971	0.01303	0.0002
1180	0.500	0.3593	0.3415	-0.017843	0.0003
850	0.360	0.2821	0.2763	-0.0058	0.0000
600	0.254	0.2068	0.2277	0.020871	0.0004
425	0.180	0.1944	0.1893	-0.005081	0.0000
300	0.127	0.1638	0.1574	-0.00642	0.0000
212	0.090	0.1306	0.1309	0.00035	0.0000
150	0.064	0.1074	0.1090	0.001637	0.0000
106	0.045	0.091	0.0907	-0.000267	0.0000
75	0.032	0.073	0.0756	0.002552	0.0000
Sum of squared errors					0.0010
φ		0.4688			
γ		0.5293			
β		5			

Table 3. The breakage function values for the microwave treated -3350+2360 µm fraction.

Particle size (µm)	Relative particle size	Breakage function		Error	Squared error
		Experiment	Model		
2360	1	1	1	0	0.0000
1700	0.720	0.4242	0.4407	0.01652	0.0003
1180	0.500	0.297	0.2719	-0.0251	0.0006
850	0.360	0.2064	0.2056	-0.0008	0.0000
600	0.254	0.1429	0.1595	0.0166	0.0003
425	0.180	0.1272	0.1253	-0.0019	0.0000
300	0.127	0.1033	0.0985	-0.0048	0.0000
212	0.090	0.0768	0.0775	0.00067	0.0000
150	0.064	0.0598	0.0610	0.00121	0.0000
106	0.045	0.0484	0.0480	-0.0004	0.0000
75	0.032	0.0355	0.0378	0.0023	0.0000
Sum of squared errors					0.0012
φ		0.4090			
γ		0.6904			
β		5			

more significant in coarse fractions in which the effect of microwave treatment is more obvious. Studying the γ in treated and untreated samples showed that microwave treatment increases this parameter to 30%. Also, the results for the Band ball mill work index showed that when time of microwave radiation was increased to 4 min, the amount of work index decreased from 12.46 kWh/t to 6.45 kWh/t (a 48% decrease).

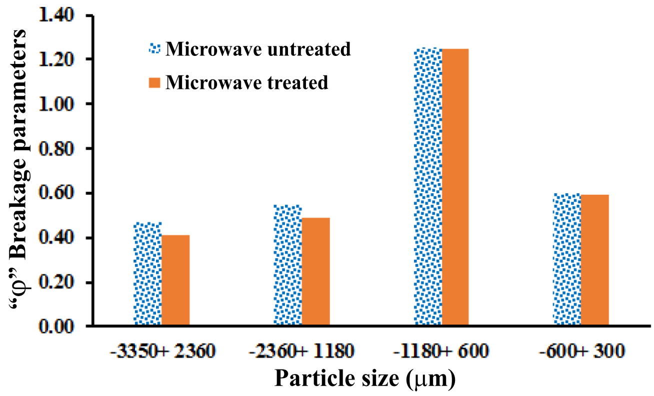

Fig. 9. "φ" Breakage distribution parameter for different fractions for treated and untreated samples.

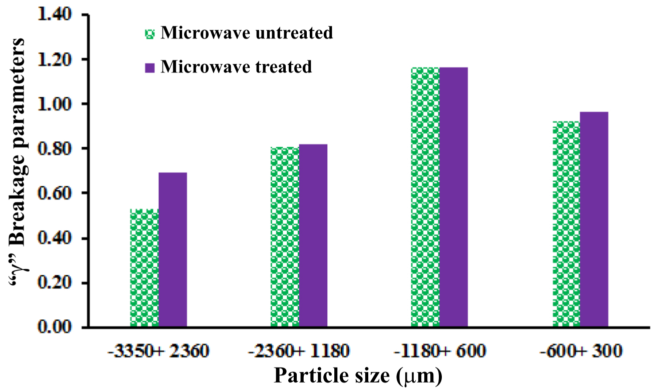

Fig. 10. "γ" Breakage distribution parameter for different fractions for treated and untreated samples.

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