

Effect of Microwave Pretreatment on Grinding of Iron Ore

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The effect of microwave radiation on grinding of an iron ore was investigated. Significant reductions in Bond work index are demonstrated for microwave treated samples. These reductions are sensitive to the power applied in relation to the exposure time of the mineral to microwaves. The best result was obtained with an exposure time of 3 min, achieving a reduction of 41 % in Bond work index. The grinding kinetics shows that there is a significant effect on the production of fines for the treated sample, since for each grinding time tested, the distribution of passing cumulative size was higher respect to the sample non-treated, particularly for short times. The results obtained indicated important improvements in the size reduction, due to microwave energy, produced microfractures intergranular and transgranular level, caused by differential heating and subsequent thermal expansion between mineral phases absorbing and transparent to microwave radiation. These changes are supported by analytical studies of X-ray diffraction and scanning electron microscopy. It was confirmed that microwave pre-treatment has a significant positive effect on the efficiency of the energy applied to the grinding of iron ore studied.

Keywords: Microwave energy, Grinding, Bond work index, Grinding kinetic.

INTRODUCTION

Comminution is recognized as highly energy-demanding process. For the finer product size more input energy is required. Enhancement of energy efficiency could be achieved in different ways. According to Wang and Forsberg [1], there are several development trends leading to more efficient comminution: (a) Development and application of new mills/classifiers, (b) Adjustment of bead characterization in stirred bead mills, (c) Hybrid comminution systems with roller-press and media mills, (d) Assisted methods of comminution (grinding aids, microwave-assisted comminution, ultrasound-assisted comminution) and (e) Simulation.

Microwave energy is a non-ionizing electromagnetic radiation with frequencies in the range of 300 MHz to 300 GHz. Microwave frequencies include three bands: the ultra high frequency (UHF: 300 MHz to 3 GHz), the super high frequency (SHF: 3 GHz to 30 GHz) and the extremely high frequency (EHF: 30 GHz to 300 GHz).

Microwaves processing of materials are a new, powerful and significantly different technology to process materials that may not be amenable to convectional means of processing or

to improve the performance characteristics of existing materials [2,3].

In recent years, several potential applications of microwave processing in minerals treatment have been investigated. These included microwave assisted ore grinding, microwave assisted carbothermic reduction of metal oxides, microwave-assisted drying and dehydration, microwave-assisted minerals leaching, microwave-assisted roasting and smelting of sulfide concentrates, microwave-assisted pretreatment of refractory gold concentrates, microwave-assisted spent carbon regeneration and microwave-assisted waste management [4].

Thermally assisted comminution reduces the energy required for subsequent grinding by generating thermal stresses that can cause weaknesses in the mineral matrix [5].

In the comminution process, microwave pretreated ores show a differential heating due to the different dielectric properties of the various phases present in the ore body. This differential heat in leads to fracture along the grain boundary and reduce the Bond work index of the material. As the energy required to comminution the ore is reduced. It is then imperative to understand the effect of the microwave exposure on downstream processing of the minerals themselves, such as the

magnetic properties and hydrophilic nature of the mineral particles [6].

In general most metal sulphides and oxides get heated readily when subjected to a microwave irradiation, on the other hand, common gangue minerals containing silicates and carbonates are nearly transparent to microwaves and get heated slightly.

Florek *et al.* [7] carried out a study of the effect of microwave treatment on the magnetization of iron ore minerals. It was concluded that the surface characteristics and magnetization of iron ore minerals alter after microwave radiation.

The effect of microwave radiation upon the mineralogy and magnetic processing of a massive Norwegian ilmenite ore was performed by Kingman *et al.* [8]. It has been shown that short period of exposure can cause fracture at grain boundaries, which leads to the formation of inter-granular fractures. This fracture coupled with an increase in remnant magnetization of the ilmenite mineral has been demonstrated to give rise to an increase in both concentrate grade and valuable mineral recovery. However, the study has also indicated that the process efficiency can be effected with over exposure to microwave radiation.

Kingman and Rowson [9] showed that a number of minerals, *e.g.* chalcopyrite, hematite and wolframite, not only heat readily during exposure to microwaves, but also exhibit a considerable increase in the magnetic susceptibility after being exposed to 650 W microwave radiation. Cui *et al.* [10] carried out an investigation to study the changes in magnetic properties after roasting to the different types of minerals contained in the oil sands tailings. It was observed that the magnetic susceptibility of ilmenite increased after either oxidation or reduction roasting. For hematite, reduction roasting increased its magnetic susceptibility and oxidation roasting did not seem to have any effect

Sahyoun *et al.* [11] investigated the influence of conventional heat treatment and microwave radiation on chalcopyrite. There was a significant increase in the proportion of material recovered to magnetic fraction and magnetic susceptibility with conventional heating time.

Uslu *et al.* [12] investigated the effect of microwave heating on magnetic processing of pyrite. The microwave treated pyrite samples of 0.420 mm fraction were subjected to magnetic separation at magnetic field intensities of 0.1, 0.3 and 0.5 T. It was found that pyrite was converted to such ferromagnetic minerals as pyrothite and γ -hematite and magnetic separation recovery was improved after microwave treatment.

Znamenackova *et al.* [13] showed that after 10 min pretreatment of weakly paramagnetic ore in a microwave oven with maximum power of 900 W, essential change in the magnetic properties of the ore samples occurred and after 15 min, a rapid increase of magnetic susceptibility value was observed, showing the intensive decomposition of siderite.

Waters *et al.* [6] investigated the effect of microwave radiation on the magnetic properties of pyrite. After treatment with a conventional multimodal reactor (2.45 GHz and 1900 W) for 120 s, the recovery of pyrite in the magnetic fraction after separation increased from 8 % (wet) and 25 % (dry) to greater than 80 % for both process streams.

Compared to conventional heating methods, the major advantages of microwave heating for industrial processing are rapid heat transfer, volumetric and selective heating, compactness of equipment, speed of switching on and off and pollution-free environment [14,15].

Comminution or rock breakage, accounts for 30-70 % of the power draw of a typical plant and for 20-50 % of the capital cost [16]. It is thought that as much as 3 % of the electrical energy generated in the US and as much as 5 % globally is consumed by comminution [17]. However, the comminution process is only 1 % efficient in terms of the energy required to generate new surfaces. Microwave pretreatment of ores, as an alternative potential technology to bring the comminution cost down, has attracted the attention of the researchers in the field of mineral processing around the world.

Kingman *et al.* [4] observed that the rapid heating of ore minerals in a non heating gangue generated thermal stresses that could cause weaknesses in the mineral matrix. This observation has been extended in Kingman and Rowson [18] where significant reductions of up to 90 % of the work index were reported for microwave treated massive Norwegian ilmenite ore in conjunction with increases in concentrate grade and valuable mineral recovery.

The objective of this work is to evaluate the effect of pretreatment with microwave radiation on the grinding of iron ore through studies, X-ray diffraction (XRD), scanning electron microscopy (SEM), grindability studies, simulation studies with MinProSim, 2.1. Program.

EXPERIMENTAL

Material and source of electromagnetic energy: The sample used in the development of this study corresponds to oxide ore from Guerrero-Mexico, with hematite (Fe_2O_3) being the main mineral phase.

The study sample was obtained from a batch of ore size - 3.5" of about 100 kg, subsequently prepared through different size reduction steps in combination with sampling stages, for mass sample and particle size required to perform different analytical studies and experimental tests. Preparation procedure was the same for all samples, so that the method of preparation would not affect grindability study. The assaying was 36 % Fe, 6.9 % Ca and 1 % Mg. It is noted that the main element is Fe, that is distributed mostly in hematite (Fe_2O_3) and magnetite (Fe_3O_4) in lesser extent.

For this study, a multi-mode microwave oven is used, with a variable power output 100 to 1800 W; with 2.45 GHz frequency, irradiation tests were performed with sample mass, 250 g; with different sizes and irradiation times using the maximum output power of the oven.

Characterization: The crystal structures besides the morphology, distribution and size of the mineral phases in the sample before and after pretreatment with microwave radiation, were obtained using following analytical techniques, X-ray diffraction and scanning electron microscopy, through the following equipment: Bruker brand - D8 Advance, model Da Vinci and scanning electron microscope - brand JEOL, model JSM - 6610LV, respectively.

Determination of effect of microwave energy on grindability: The effect of microwave radiation upon the grindability of iron ore was quantified by Berry and Bruce method [19]. This method requires the use of an ore of known grindability. The reference ore is ground for a certain length of time and the power consumption recorded. If an identical weight of test ore is ground for the same time period then the power consumption will be equal to that of the reference ore. Then if 'r' is the reference ore and 't' the ore under test, from Bond equation (eqn. 1) obtain the mathematical relationship for calculating the W_i (eqn. 3) for different operating conditions to be studied in this work.

$$W = \frac{10W_i}{\sqrt{P}} - \frac{10W_i}{\sqrt{F}} \quad (1)$$

where; W_i = Bond work index, P = 80 % passing size of product, F = 80 % passing size of feed, W = work input per short ton, then for the two minerals we will have:

$$W_r = W_t = W_{i_r} \left(\frac{10}{\sqrt{P_r}} - \frac{10}{\sqrt{F_r}} \right) = W_{i_t} \left(\frac{10}{\sqrt{P_t}} - \frac{10}{\sqrt{F_t}} \right) \quad (2)$$

Therefore,

$$W_{i_t} = W_{i_r} \frac{\left(\frac{10}{\sqrt{P_r}} - \frac{10}{\sqrt{F_r}} \right)}{\left(\frac{10}{\sqrt{P_t}} - \frac{10}{\sqrt{F_t}} \right)} \quad (3)$$

Comparative grinding tests were conducted on representative 1 kg samples of ore. Each sample had an identical size distribution.

To quantify changes in grindability a ball mill of 0.66 × 0.9 ft, was used; for which were calculated and established operating parameters: balls-charge filling volume of the mill (40 %), % speed critical (75), % solids (60), feeding size (- 6 #). Grinding was continued for 3 min. After the required grinding period, the 80 % passing size of the ground product was determined by sieve analysis. Samples were irradiated for times varying between 60 and 240 s.

Method for determination of Bond work index (W_i) (kWh/t): This test is performed to determine the resistance of the reference sample to grinding, said resistance expressed by the energy requirement (kWh) to grind one ton of ore to a

given reduction ratio. To perform this test mill 12" × 12" is used, with a load of 20,125 kg balls working with a feed size -6 # (3350 μ m).

With the information obtained in this test, work index (W_i) are obtained by the following relationship.

$$W_i = \frac{44.5}{(P_i)^{0.23} (Gbp)^{0.82} \left(\frac{10}{\sqrt{P_{80}}} - \frac{10}{\sqrt{F_{80}}} \right)} \quad (4)$$

where: W_i = Bond work index (Kw h/t short). F_{80} and P_{80} = 80 % passing size of feed and 80 % passing size of product respectively. P_i = Particle size passing mesh opening reference. Gbp = grams produced per revolution.

Grinding kinetics: A serie of grinding tests was performed with the best conditions obtained in the above test, to sample before and after pre-treatment with microwave radiation, varying the grinding time (3, 5,10 and 15 min) to determine the degree of size reduction, through the particle size distribution obtained for each time used.

RESULTS AND DISCUSSION

The X-ray diffraction patterns of the samples before and after being microwave treated are shown in Fig. 1. Analyzing Fig. 1a and 1b, a generalized increment in the intensity of the characteristic peaks of the phases present in the treated sample, respect to the phases of the non-treated sample; which is due to increased crystallinity by a rearrangement of the crystalline structure, also new phases as goethite and maghemite are produced according to the degree of oxidation of magnetite generated in the microwave process.

Fig. 2 shows the SEM images of sample under investigation before and after being microwave treated, where the Fig. 2a and 2b, shows the types, shape, size and distribution of the mineral phases in the ore sample, while Fig. 2c to 2g show the effect of radiation as the particle size varies, where appreciate micro-fractures weaken the strength ore in grinding and finally generate an energy saving in this operation.

Bond work index (W_i) (kWh/t): The Bond work index for the reference sample (W_{i_r}) was 8.72 kW h/t short (9.6 kW h/t).

Grindability study: Fig. 3 shows the effects of microwave radiation time on the Bond work index of iron ore. The same

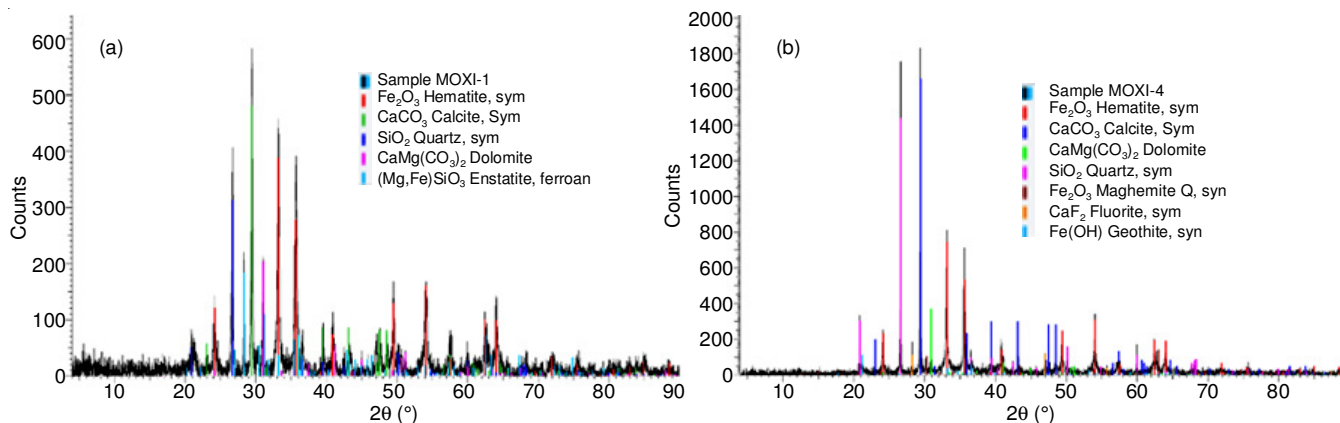


Fig. 1. XRD pattern of the sample under study, for microwave non-treated (a) and microwave treated iron ore (b)

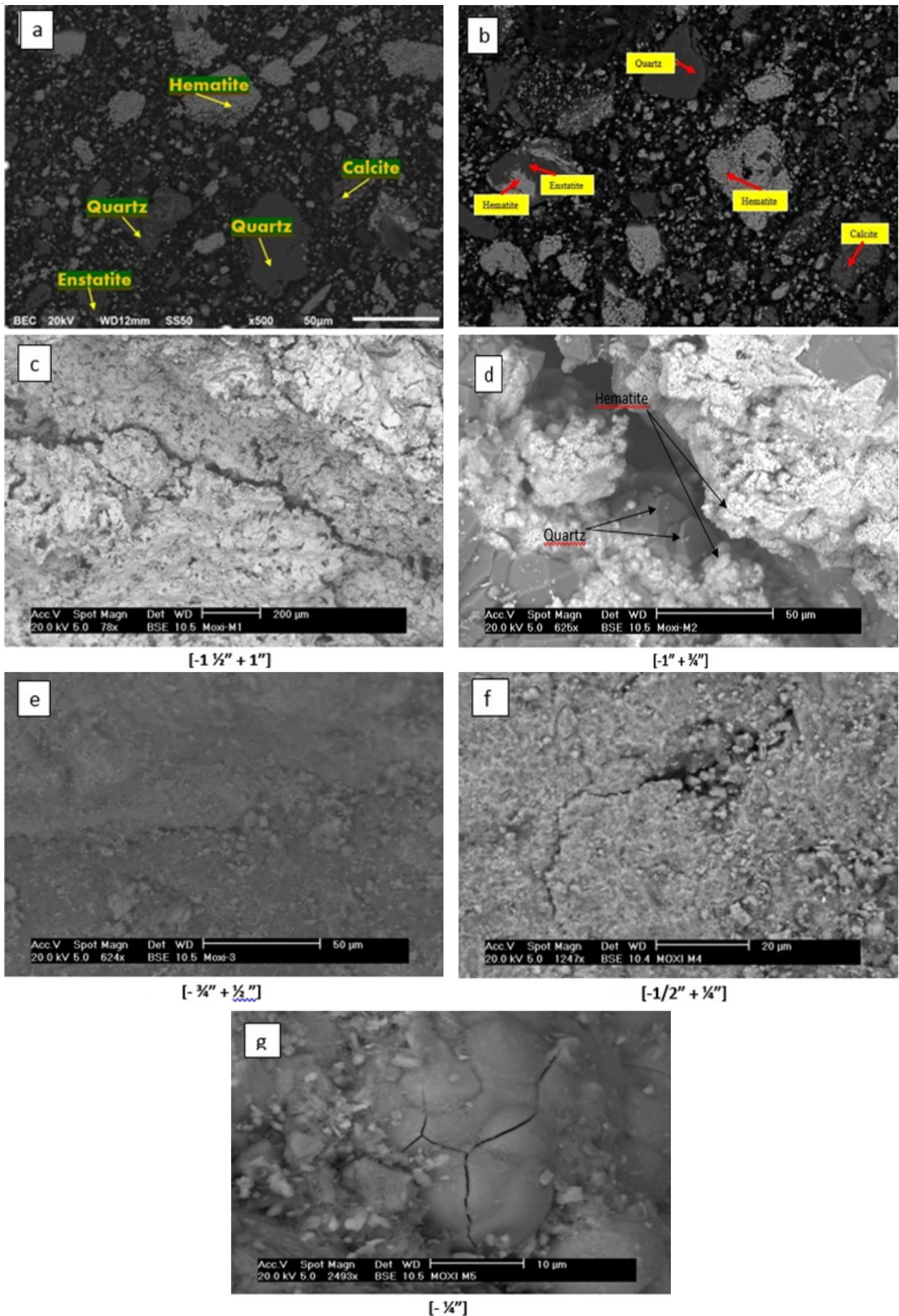


Fig. 2. SEM images; microwave non-treated (a and b) and microwave treated (c-g)

figure clearly shows that microwave treatment had a significant effect upon the Bond work index of this ore. Initially for microwave exposure times of 1 and 2 min, the effect was similar in both cases having a decrease of Bond work index of the ore under test (W_i) around 24 %. The most important effects has been treatment period where Bond work index is shown to reduce 41 % (from 9.6 kWh/t to 5.7 kWh/t) after 3 min irradiation. The reason for this larger reduction in Bond work index after microwave treatment. It is due to the mechanism of the process which consists in the differential heating and therefore, different expansion of the minerals within the mineral matrix. This differential expansion causes significant microfractures (Fig. 2c to 2g). This can have several benefits: firstly, grinding energy can be reduced and secondly increased liberation of mineral species resulting in an increase in metallurgical results. When it increases the microwave exposure time to 4 min, the value of Bond work index of the ore under test (W_i) returns values for times of 2 and 3 min, which means that for long exposure times, the results are less favourable in saving energy used in grinding of iron ore. The reason for this change is the production of new mineral phases such as goethite and maghemite (Fig. 1b) due to the oxidation of the magnetite and knowing that their mineralogical, physical and chemical properties are different from those that gave rise to them.

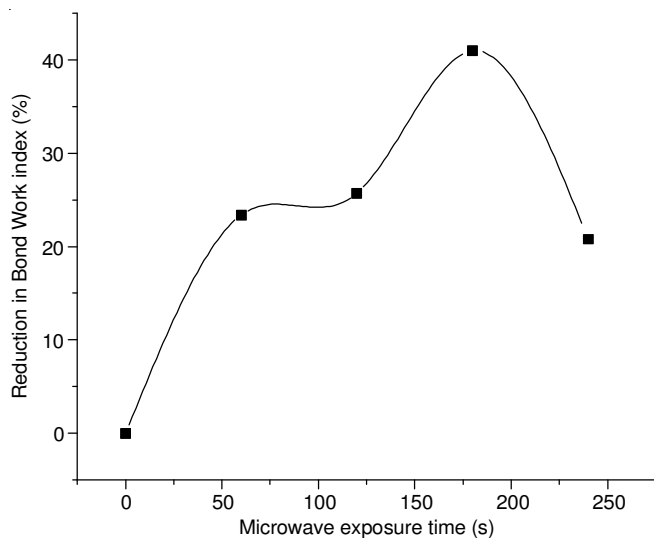


Fig. 3. Effect of microwave exposure time on Bond work index of iron ore

Simulation studies: They were performed through simulation program MinProSim, 2.1 supplied by Laboratory Simulation, Metallurgy Institute- Universidad Autónoma de San Luis Potosí, México, for estimate the increase of grinding capacity of a given mill of known dimensions and operating conditions determined, considering the percentages of reduction obtained in the Work Index of iron ore. The results shown in Fig. 4, indicates an increase in plant throughput in the same direction and approximately in the same proportion as the percentage reduction of Bond work index. It should be borne in mind that this projection corresponds to a specific case only.

Grinding kinetics: Fig. 5 shows the comparative size distribution throughout the size range tested (6 to 400 mesh). The graphs reflect a constant dominance of fine particles generated in microwave exposed sample over the non-microwaved

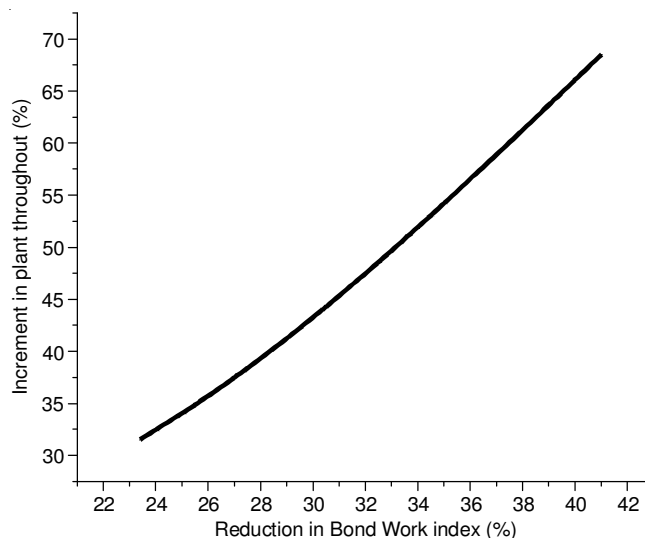


Fig. 4. Estimate the increment plant throughput rate with percent reduction in Bond work index, using MinProSim 2.1

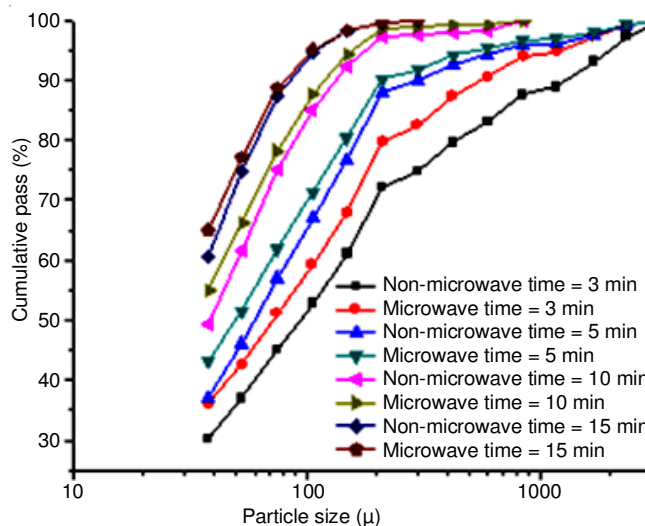


Fig. 5. Variation product size distribution with time of grinding for microwave non-treated and treated sample

sample in the grinding. The observation reveals that less ball mill grinding time is desired to generate same cumulative percentage passing value (P_{80}) for every mesh. This signifies that the microwaved sample it is affected in its structure by generating microcracks and fissures developed intergranular and transgranular level and which weakens their resistance to fracture resulting in the reduction of Bond work index (Kwh/t). The operating conditions for this test are the same as mentioned above, for a sample mass of 1 kg and with a microwave exposure time of 3 min.

Based on the results obtained it can be defined that there is a potential use of microwave energy as a pre-treatment method to achieve greater efficiency in energy use in grinding, considering technological innovations in the microwave generator equipment and essentially the mineralogy of the sample to be pretreated with this type of electromagnetic radiation.

Conclusions

- The sample studied suffered physical, chemical and mineralogical changes as a result of the microwave radiation

process, these changes were supported by studies of X-ray diffraction and scanning electron microscopy.

- Reductions in Bond work index ($W_i - kWh/t$) were found to be sensitive to applied microwave radiation time, achieving the greatest reduction (41 %) with a time of 3 min.

- The results in grinding kinetics show a higher value of cumulative per cent mass passing each size used to the treated sample compared to the non-treated with microwaved for each used time, indicating that the process of microwaved irradiation decreased the resistance to fracture in the grinding operation. Therefore states that the use of microwave radiation achieves greater efficiency in energy applied to the grinding.

- The saving energy in grinding by the percentage of reduction in the Bond work index due to pretreatment with microwave energy, resulted in an increase similar in grinding capacity, according to the MinProSim 2.1, Simulation Program.

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Consejo Nacional de Ciencia y Tecnología (Conacyt) and Universidad Autónoma de Zacatecas(UAZ) supported this work. Universidad Autónoma de San Luis Potosí (UASLP) through Doctorado Institucional de Ingeniería y Ciencia de Materiales (DICIM) and Laboratorio de Simulación del Instituto de Metalurgia, for opening the doors to pursue a Doctorate in Engineering – Materials and to every one for making this work possible.

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