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Microwave Pretreatment on Copper Sulfide Ore: Comparison of Ball Mill Grinding and Bed Breakage Mechanism

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Abstract

The aim of this paper is to assess the effect of microwave treatment on copper sulfide ore for ball milling and bed breakage. Liberation analysis of ground products in eight size fractions showed that microwave pretreatment liberated more chalcopyrite particles from the bed breakage product than the ball mill product. In bed breakage, microwave pretreatment caused a 2% increase in chalcopyrite liberation on average. Microwave pretreatment produces more elongated particles in ball milling and more rounded particles in bed breakage, respectively. Microwave pretreatment produces coarser chalcopyrite particles with a wide range of sizes. This effect is more significant in bed breakage compared with ball milling. Micro-flotation test results showed that in ball milling, treated and untreated materials have similar metallurgical performance and there is a little difference (1%) between the average Cu recovery values. However, in bed breakage, the average recovery for the treated materials is about 5% higher than the untreated materials.

Keywords Microwave treatment · Bed breakage · Ball milling · Particle shape · Liberation · Flotation

1 Introduction

The mechanism of breakage in a tumbling mill is a combination of impact, abrasion, and attrition. Each breakage mechanism produces a powder of different particle size distributions and shapes [1]. In equipment such as High-Pressure Grinding Roller mills (HPGR) and Vertical Roller mills (VRM), particles are ground by compression in a bed of particles. This type of breakage is called bed breakage [2].

The effects of microwave treatment have been investigated on the strength reduction of rocks, the results show that selective heating induces microcracks on the grain boundaries within the rock structure. This is favorable for application in excavation and comminution [3–7].

The effect of microwave radiation upon grinding in tumbling mills such as rod mills and ball mills has been studied by many researchers, which show the grindability and minerals liberation increase after microwave pretreatment. However, a small initial increase or adverse effects on the recovery of the subsequent separation process were reported [8–18]. Also, the potential industrial application of microwave pre-treatment processes for beneficiation of ores was investigated at The University of Nottingham. A pilot-scale microwave treatment system was designed, constructed, and commissioned for treating porphyry copper ores at throughputs of up to 150 tph. The materials were treated by a single applicator (1 × 100 kW, 100 tph) or a Dual applicator (2 × 100 kW, 150 tph). The results showed that two single-mode applicators in series yielded equivalent or better power consumption and metallurgical performance compare to a single mode applicator. They claimed that by fully integrating the applicator with a materials handling system, a reliable and continuous process can be achieved and the system can be used to treat a range of porphyry copper ores [19, 20].

Many researchers studied the bed breakage mechanism and concluded that it can increase the liberation of minerals as a result of the preferred fracture occurring at the grain boundary [21–26]. However, some studies have shown that

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bed breakage did not improve the liberation of minerals compared to impact breakage [27].

To the authors' knowledge, there are only two published papers that presented the effects of microwave pretreatment on bed breakage [28, 29]. Ali and Bradshaw examined the effect of microwave treatment on mineral liberation through the simulation of single-particle and confined particle bed breakage of bonded-particle models. Their results showed that microwave treatment altered the fracture pattern and the degree of liberation. In these researches, the bed breakage and microwave treatment were simulated by the discrete element method using the commercially available software PFC2D. However, verification experiments with ore samples were not carried out as part of these studies.

The aim of the current research is to conduct experimental studies to compare the effect of microwave pretreatment and its influence on ball mill grinding and bed breakage for the fist time. This is performed on copper sulfide ore in terms of mineral liberation, particle shape, and floatability.

2 Exprimental

2.1 Materials

Copper ore samples were collected from the Qaleh-Zari vein copper mine which is located in Southern Khorasan Province, Iran. The samples were crushed to 100% passing 3.36 mm. Figure 1 shows the particle size distribution of the crushing product. The crushing product was riffled into two samples for bed breakage and ball milling.

A representative sample was identified by X-ray diffraction analysis (XRD, Stoe company, model STADIP), as shown in Fig. 2. The results indicate that chalcopyrite, galena, and quartz are the main minerals in the sample. Also, the chemical composition was determined by total x-ray fluorescence (TXRF, GNR company, model TX2000). Table 1 shows the chemical composition of the sample.

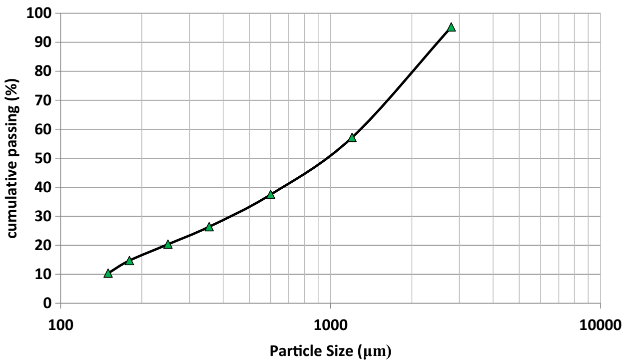
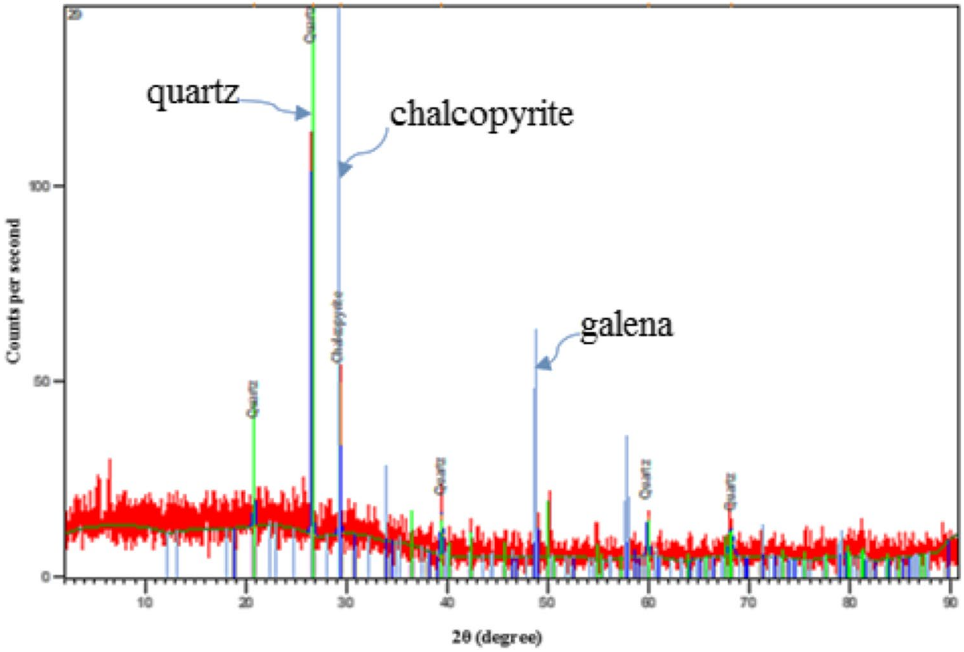


Fig. 1 Particle size distribution of the crushing product

Table 1 The chemical composition of the sample

Element	Pb	Cu	Fe	S	Si
%	5.92	7.71	10.83	25.50	50.04

Fig. 2 XRD pattern of the sample



2.2 Microwave Treatment

For each grinding mechanism (bed breakage and ball milling), one sample with 800 g weight was treated in a domestic multi-mode microwave oven for 10 min (Samsung, model ST656W, 2.45-GHz, 900 W rated power). In this study, the effect of the microwave heating was investigated and the type of oven and its economic feasibility were not considered. Sample geometry factors such as volume, surface area, and location in the oven are important in the magnitude and uniformity of power absorption [30]. To minimize these effects, in all radiation experiments, the surface area and thickness of the powder spread on the tray were kept constant, and the tray was located in the same central position.

2.3 Bed Breakage Tests

The bed breakage tests were carried out in a piston-die cell with a cylindrical diameter of 100 mm, a piston diameter of 90 mm, and a height of 200 mm (Fig. 3). In this test, a small sample of materials was placed in a cylindrical chamber and then the piston was forced on the materials. This test was extensively used in the literature to model the bed-breakage grinding [21, 22, 26, 31, 32]. A 2000-kN hydraulic press is used in this project. For each test, 350 g of the sample was poured into a cylinder and pressed to 1950 kN. The pressed cake was collected from the cylinder and passed through a 250-μm sieve. The remaining materials on the sieve back to the cylinder and pressed again, this was repeated until the entire materials passed through a 250-μm sieve.

2.4 Ball Mill Grinding Tests

Ball mill grinding tests were performed in a 20 cm × 20 cm stainless steel laboratory rotary mill. It was operated at a constant speed of 85 rpm (84% of the critical speed). The diameter of stainless-steel balls is 16–42 mm with a total weight of 8.79 kg and ball voids of 43.33%, which is 25% of the total volume of the mill. The fraction of the ball charge filled by powder was 0.27. The ground product was passed through a 250-μm sieve. The remaining materials on the sieve back to the mill and ground again, this was repeated until the entire materials passed through a 250-μm sieve.

Fig. 3 The piston-die cell under pressure

2.5 Mineral Liberation

The bed breakage and ball mill products were dry sieved into eight size fractions (− 250 + 177 μm, − 177 + 150 μm, − 150 + 106 μm, − 106 + 75 μm, − 75 + 63 μm, − 63 + 44 μm, − 44 + 37 μm, − 37 μm). Figures 4 and 5 show the particle size distributions of the bed breakage and ball mill products, respectively. Polished sections were prepared from the eight-size fractions and the liberation degree of chalcopyrite was determined for each size fraction by a Zeiss Axioplan 2 optical microscope. The procedure was based on counting all liberated and locked chalcopyrite particles.

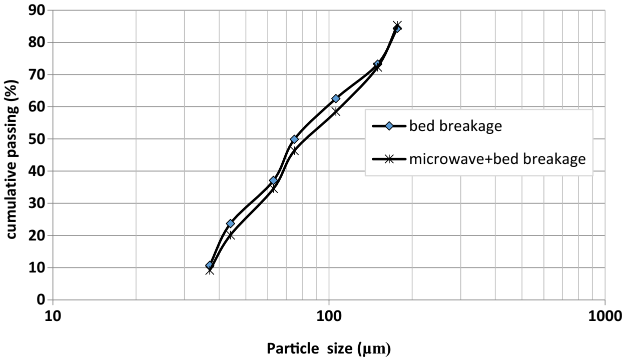


Fig. 4 Particle size distribution of bed breakage

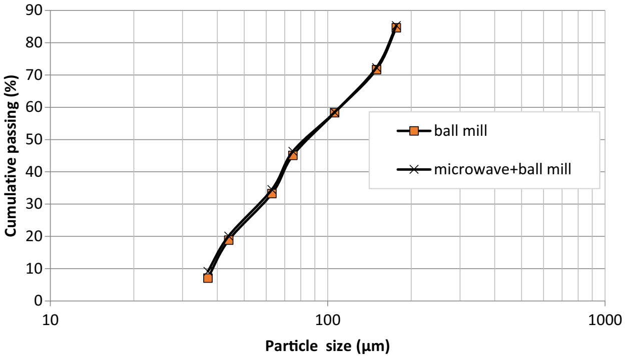


Fig. 5 Particle size distribution of ball milling

2.6 Particles Shape

The particle shape is known to affect the flotation behavior significantly as indicated in fairly recent qualitative studies [33]. Polished sections were prepared from the breakage products and backscatter images were taken by SEM (TESCAN, model FESEM). In each polished section, approximately 80 chalcopyrite particles were manually (for more accuracy) recognized by EDS analysis (Fig. 6). Given the abundance and distribution of chalcopyrite, this is a statistically significant population. The shape properties of particles such as circularity, roundness, and aspect ratio were determined with sufficient accuracy using the microscopic image analysis software (MIP4, Nahamin Pardazan Asia).

The shape parameters, namely circularity, roundness and aspect ratio can be defined as follows [34]:

$$Circularity = \frac{4\pi S}{P^2} \tag{1}$$

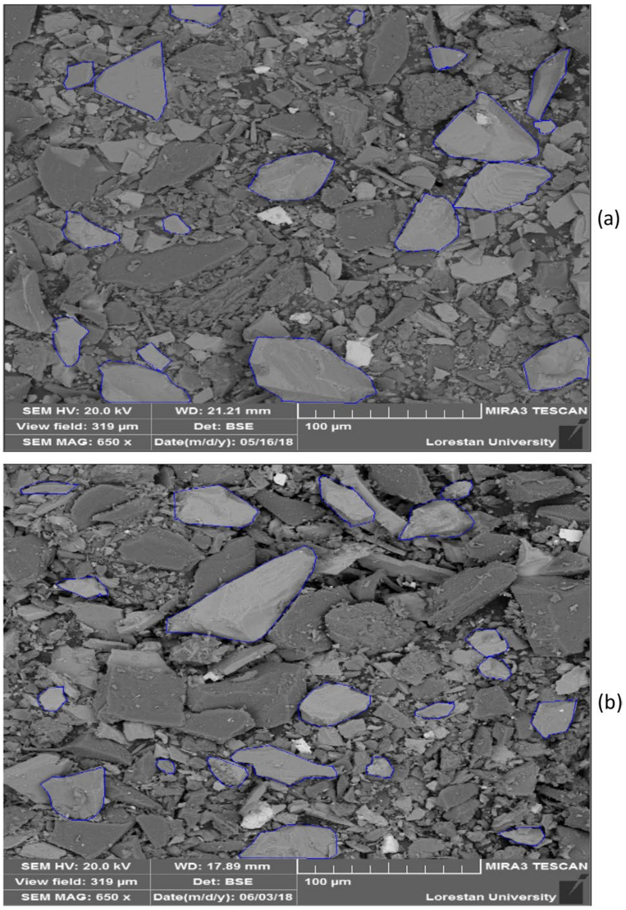


Fig. 6 SEM images: ball mill product (**a**) and bed breakage product (**b**) products. Chalcopyrite particle are marked with blue border

$$Roundness = \frac{4S}{\pi D^2} \tag{2}$$

$$Aspect\ Ratio = \frac{L}{W} \tag{3}$$

where L is length, W is width, and S is cross-section area. L and W are the long and short sides of the smallest rectangle that surrounds the particle, respectively. The diameter (D) is the longest segment inscribed in the particle. S and P are the area and perimeter of the particle, respectively (see Fig. 7).

The factor of circularity is a combination of geometric shape and surface smoothness. Assuming a circularity value of 1 for a sphere with surface asperities below the level of detection, circularity values > 1 are considered for a sphere with rough surfaces and particles of any other shapes. The aspect ratio depends on the particle orientation. Given their orientations, particles of arbitrary shapes may exhibit aspect ratios smaller or higher than unity (reserved for spheres that have an aspect ratio of 1). Both low and high values of the aspect ratio indicate irregularity in particle shape. Roundness describes the sharpness of particle corners and edges, which corresponds to the ratio of the mean curvatures at the corners of the particle to the diameter of the largest circle surrounding the particle. The maximum roundness of 1 can be reached in the case of perfectly circular shapes [34].

2.7 Micro Flotation Experiments

The micro flotation tests were carried out in a 150 cm^3 Hallimond tube (Fig. 8). In each test, 3 g of each size fraction

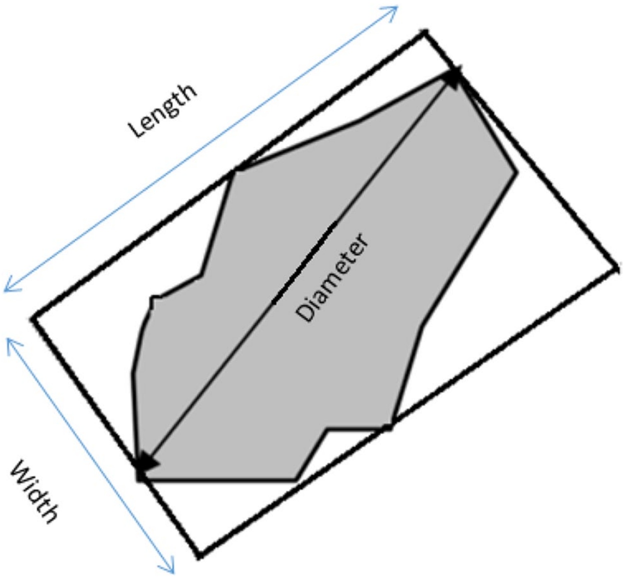


Fig. 7 Parameters of particle shape

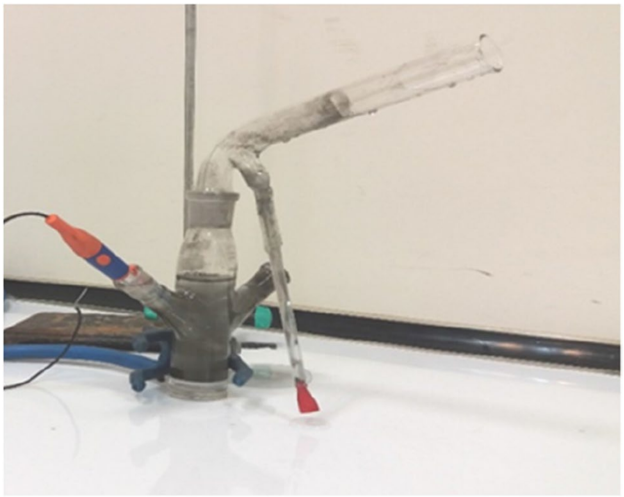


Fig. 8 Hallimond tube

was used by adding it to the double-distilled water (DW) and conditioning the mixture for 5 min. After this period, the collector (potassium amyl xanthate) and frother (methyl isobutyl carbinol (MIBC) with a concentration of 10 mg/L were added to the suspension. The pH was adjusted around 10 and a second conditioning stage of 15 min was given to the suspension. The prepared pulp was then transferred to the Hallimond tube, where flotation was carried out for 4 min. Each experiment was repeated three times. At the end of the experiment, the concentrate and tailings were collected, dried and, analyzed by atomic absorption spectrometry (Agilent model AA240FS) to estimate the amount of copper.

3 Results and Discussion

Figure 9 shows the effect of microwave treatment on chalcopyrite processing. It is shown that in ball milling, microwave pretreatment has a little negative impact on chalcopyrite liberation at all size fractions. However, in bed breakage, microwave pretreatment increased chalcopyrite liberation, especially at coarse size fractions. In bed breakage, microwave pretreatment on average caused a 2% increasing in chalcopyrite liberation. This is a significant improvement in grinding. Microwave heating causes thermal stress fracturing along grain boundaries and this could increase grindability of ores and mineral liberation as well [12].

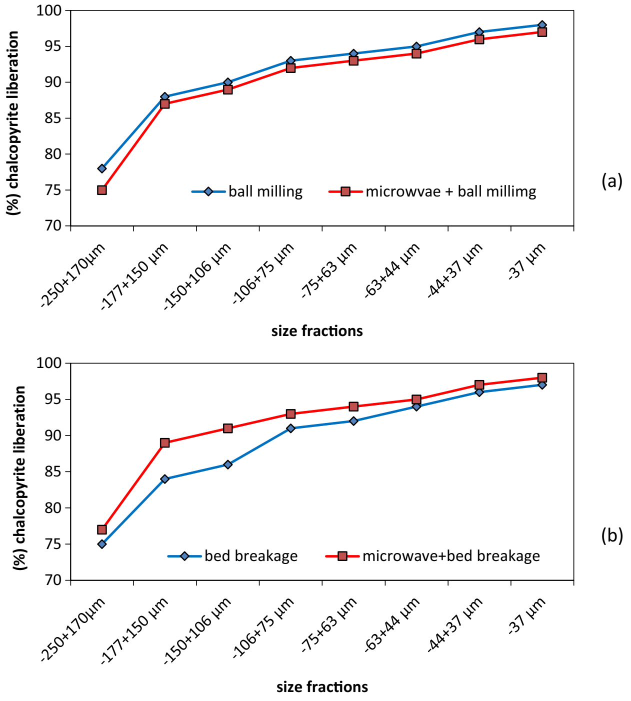


Fig. 9 Effect of microwave treatment on chalcopyrite liberation: ball milling (**a**), bed breakage (**b**)

Figure 10 shows the average particle shape factors. The data in Fig. 10 shows that in ball milling, microwave pretreatment has no significant effect on circularity and roundness of particles, but the aspect ratio relatively increased by about 3.6%. It means that in ball milling, microwave pretreatment causes the production of elongated particles. As in bed breakage, microwave pretreatment increased circularity by 1.4% and roundness by 5.06%, this decreased aspect ratio. It can be concluded that in bed breakage, microwave pretreatment causes the production of more rounded particles.

For each breakage condition, more than 160 particles were counted in the SEM images and their circular diameters were measured. Figure 11 shows the numerical percentage of particles and Fig. 12 indicates the corresponding average size and the standard deviation. The figure also reveals the similarity ($R^2 = 0.91$) between the microwave treated and untreated materials processed in ball milling. The

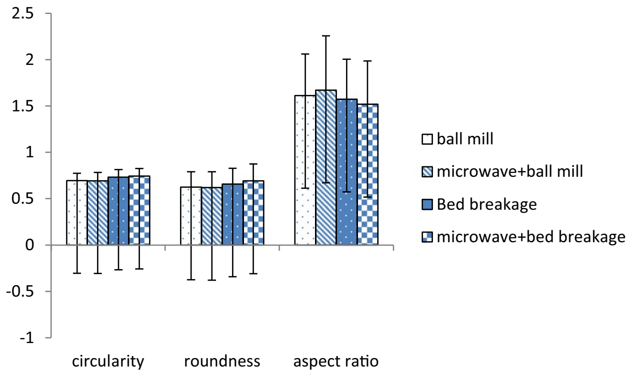


Fig. 10 Comparison of the shape properties of chalcopyrite particles for different breakage (Error bars show the standard deviation)

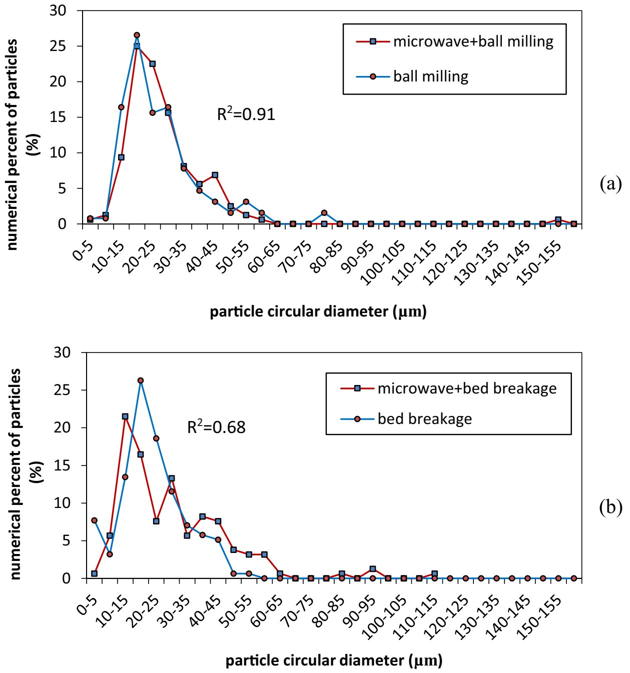


Fig. 11 Numerical percentage of chalcopyrite particles in ball milling (**a**) and bed breakage

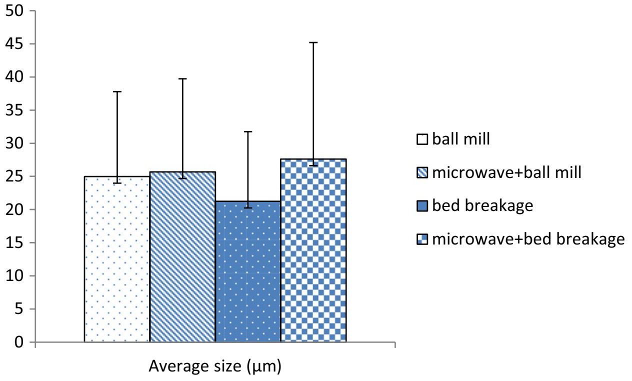


Fig. 12 The average particle size and standard deviation of chalcopyrite particles for different breakage (Error bars show the standard deviation)

measurements also show that bed breakage offers a lower similarity ($R^2 = 0.68$). Figure 11 and the data presented in Fig. 12 show that microwave pretreatment produces coarser chalcopyrite particles with a wide range of sizes (by considering standard deviation values). The chalcopyrite mineral grains have most likely been shielded from the fracture by the inter-granular cracking due to microwave pretreatment, resulting in a larger product. It is in close accordance with the results presented in earlier studies [12, 35]. This effect is more significant in bed breakage compared to ball milling. Likewise, Fig. 4 shows that bed breakage with and without microwave treatment produces d_{50} of 85 µm and 75 µm, respectively, although both were ground to 100% passing 250 µm.

Figure 13a shows the copper recovery of micro-flotation experiments for ball mill products. It is shown that treated and untreated materials have similar metallurgical performance and there is a little difference (1%) between the average Cu recovery values. This difference can be attributed to the greater liberation of chalcopyrite grains in the untreated material (see Fig. 9a).

Figure 13b shows the copper recovery of micro-flotation experiments for bed breakage products. It is shown that treated materials produce higher recoveries than untreated materials in the case of fine fractions and lower recoveries in the case of coarse size fractions. However, the average recovery for the treated is about 5% higher than the untreated materials. The better metallurgical performance of the treated material compared to the untreated material can be attributed to two reasons. Firstly, chalcopyrite particles were more exposed in the treated materials than in untreated materials (see Fig. 9b). Secondly, untreated materials were finer than the treated materials (see Figs. 4 and 11b). The percentage of particles of − 37 µm and − 20 µm in the untreated material was higher than their treated counterparts. The particle shape properties can also explain the different recoveries. The particles of the treated materials have higher circularity and roundness than untreated materials. Schmidt and Berg (1996) investigated the role of particle

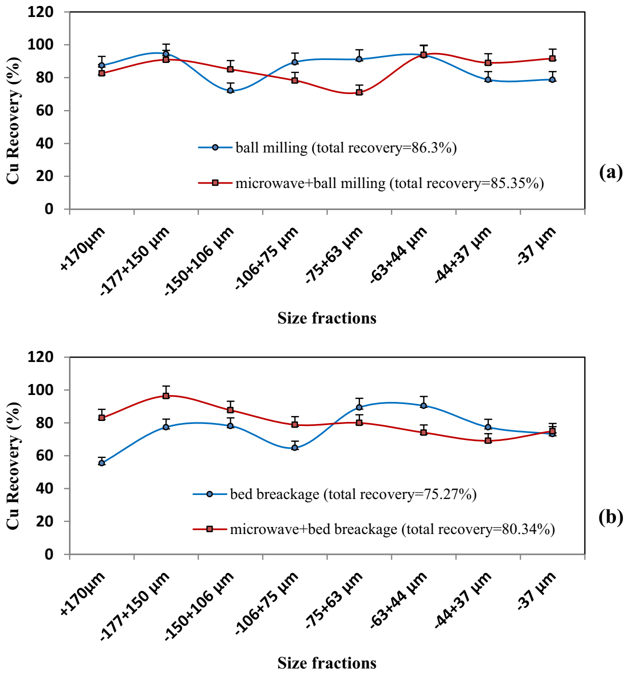


Fig. 13 Cu flotation recovery for the ball mill (**a**) and bed breakage (**b**), (Error bars show the standard deviation)

shape on flotation using model particles of spherical and the disc shapes. Their results revealed that spherical particles float better than the disc or platy particles. Although flow defects spheres away from bubbles, they still attach to the bubble surface. Normally, due to the very short contact time discs colloid with the bubble edge-on, immediately skip off, and rarely attach to bubbles. Alternatively, discs turn to the side as they approach the bubble but seldom attach due to the large thin film drainage area [36]. It was also found that large discs have always a lower probability of attachment than equivalent spheres due to the higher tendency of discs to bounce off the bubble surface after the collision. For smaller discs, the differences in attachment efficiency and flotation behavior between discs and spheres decrease, and collision efficiency becomes the predominant step [37]. This result may slightly differ from the results presented by other researchers that the flotation recoveries of non-spherical particles are higher than those of spherical particles. A possible explanation can be attributed to the increase of the specific surfaces of non-spherical particles, which leads to more adsorption of surfactant and hence increasing flotation recovery. Angular shape particles have much higher bubble attachment efficiency than spheres, which can be explained by the easier rupture of the ‘‘wetting film’’ [38–42].

4 Conclusion

A copper sulfide ore was crushed to 100% passing 3.36 mm, then the crushed product was ground by bed breakage or ball milling. The effect of microwave pretreatment on ball mill grinding and bed breakage was investigated in terms of mineral liberation, particle shape, and floatability.

In ball milling, microwave pretreatment has a little negative impact on chalcopyrite liberation at all size fractions, while in bed breakage, microwave pretreatment increased chalcopyrite liberation, especially at coarse size fractions. In bed breakage, microwave pretreatment caused a 2% increase in chalcopyrite liberation on average.

In ball milling, microwave pretreatment produces elongated particles, whereas in bed breakage microwave pretreatment produces more rounded particles. Microwave pretreatment yields coarser chalcopyrite particles with a wider size range (by considering standard deviation values). This effect is more significant in bed breakage compared to ball milling.

In ball milling, treated and untreated materials have similar metallurgical performance and there is a little difference (1%) between the average Cu recovery values. This difference can be attributed to the greater liberation of chalcopyrite grains in the untreated material. In bed breakage, treated materials exhibit a 5% higher recovery than untreated materials. This better metallurgical performance can be due to three reasons; chalcopyrite particles were more liberated in the treated materials compared to untreated materials, untreated materials were finer than treated materials, and treated particles have higher circularity and roundness (i.e. less aspect ratio) than untreated particles.

It can be concluded that microwave pretreatment has much more influence on bed breakage than on ball milling. In ball milling, the materials were ground in twenty minutes; much energy is available and applied to the materials, which limits the effectiveness of microwave pretreatment. However, bed breakage is a low energy process and the materials were crushed 3–4 times by the hydraulic pressure, which makes microwave treatment effective especially for coarser grains. Therefore, it can be concluded that increasing irradiation time or microwave power does not lead to a greater impact of microwave treatment on the ball milling.

Declarations

Conflict of Interest On behalf of all authors, the corresponding author states that there is no conflict of interest.

References

1. Napier-Munn TJ, Morrell S, Morrison RD, Kojovic T (1996) Mineral comminution circuits: their operation and optimisation.
2. Schönert K (1996) The influence of particle bed configurations and confinements on particle breakage. Int J Miner Process 44:1–16
3. Hartlieb P, Kuchar F, Moser P, Kargl H, Restner U (2018) Reaction of different rock types to low-power (3.2 kW) microwave irradiation in a multimode cavity. Miner Eng 118:37–51
4. Shepel T, Grafe B, Hartlieb P, Drebenstedt C, Malovyk A (2018) Evaluation of cutting forces in granite treated with microwaves on the basis of multiple linear regression analysis. Int J Rock Mech Min Sci 107:69–74
5. Hassani F, Nekoovaght PM, Gharib N (2016) The influence of microwave irradiation on rocks for microwave-assisted underground excavation. J Rock Mech Geotech Eng 8(1):1–15
6. Hassani F, Shadi A, Rafezi H, Sasmito AP, Ghoreishi-Madiseh SA (2020) Energy analysis of the effectiveness of microwave-assisted fragmentation. Min Eng 159:106642
7. Batchelor AR, Jones DA, Plint S, Kingman SW (2015) Deriving the ideal ore texture for microwave treatment of metalliferous ores. Min Eng 84:116–129
8. Kingman SW, Vorster W, Rowson NA (2000) The effect of microwave radiation on the processing of Palabora copper ore. J South Afri Inst Min Metall 100(3):197–204
9. Vorster W, Rowson NA, Kingman SW (2001) The effect of microwave radiation upon the processing of Neves Corvo copper ore. Int J Min Process 63(1):29–44
10. Kumar P, Sahoo BK, De S, Kar DD, Chakraborty S, Meikap BC (2010) Iron ore grindability improvement by microwave pre-treatment. J Ind Eng Chem 16:805–812
11. Rizmanoski V (2011) The effect of microwave pretreatment on impact breakage of copper ore. Min Eng 24(14):1609–1618
12. Javad Koleini SM, Barani K, Rezaei B (2012) The effect of microwave treatment on dry grinding kinetics of iron ore. Miner Process Extr Metall Rev 33(3):159–169

13. Marland S, Han B, Merchant A, Rowson N (2000) The effect of microwave radiation on coal grindability. Fuel 79(11):1283–1288
14. Amankwah RK, Pickles CA (2009) Microwave roasting of a carbonaceous sulphidic gold concentrate. Min Eng 22(13):1095–1101
15. Amankwah RK, Ofori-sarpong G (2011) Microwave heating of gold ores for enhanced grindability and cyanide amenability. Min Eng 24(6):541–544
16. Koleini SMJ, Barani K (2008) The effect of microwave radiation upon grinding energy of an iron ore, in *Microwave Technology Conference, Cape town, South Africa*
17. Kingman SW, Vorster W, Rowson NA (2000) The influence of mineralogy on microwave assisted grinding. Min Eng 13(3):313–327
18. Marion C, Jordens A, Maloney C, Langlois R, Waters KE (2016) Effect of microwave radiation on the processing of a Cu-Ni sulphide ore. The Canadian Journal of Chemical Engineering 94(1):117–127
19. Buttress AJ, Katrib J, Jones DA, Batchelor AR, Craig DA, Royal TA, Dodds C, Kingman SW (2017) Towards large scale microwave treatment of ores : part 1 — basis of design, construction and commissioning. Min Eng 109:169–183
20. Batchelor AR, Buttress AJ, Jones DA, Katrib J, Way D, Chenje T, Stoll D, Dodds C, Kingman SW (2017) Towards large scale microwave treatment of ores: Part 2—Metallurgical testing. Min Eng 111:5–24
21. Ozcan O, Benzer H (2013) Comparison of different breakage mechanisms in terms of product particle size distribution and mineral liberation. Min Eng 49:103–108
22. Hosten Ç, Özbay C (1998) A comparison of particle bed breakage and rod mill grinding with regard to mineral liberation and particle shape effects. Minerals engineering 11(9):871–874
23. Kellerwessel HAM (1996) High pressure particle bed comminution—state of the art, application, recent developments. E&MJ-Engineering and Mining Journal 197(2):45
24. Djordjevic N, Morrison R (2006) Exploratory modelling of grinding pressure within a compressed particle bed. Min Eng 19(10):995–1004
25. Khanal M, Schubert W, Tomas J (2007) Discrete element method simulation of bed comminution. Min Eng 20(2):179–187
26. Kalala JT (2011) Using piston die tests to predict the breakage behavior of HPGR, in 5th International Conference Autogenous and Semi- Autogenous Grinding, September 2011.
27. Johnson N, Vizcarra TG, Wightman EM, Johnson NW, Manlapig EV (2018) The effect of breakage mechanism on the mineral liberation properties of sulphide ores. Min Eng 23(5):374–382
28. Ali AY, Bradshaw SM (2011) Effect of microwave treatment on mineral liberation through bonded particle modelling and simulation of single particle compression, in Computational Modelling ‘11
29. Ali AY, Bradshaw SM (2011) Confined particle bed breakage of microwave treated and untreated ores. Min Eng 24(14):1625–1630
30. Barani K, Koleini SMJ, Rezai B, Khodadadi A (2012) Effect of sample geometry and placement on iron ore processing by microwave. Advanced Materials Research 488:841–846
31. Esnault VPB, Zhou H, Heitzmann D (2015) New population balance model for predicting particle size evolution in compression grinding. Min Eng 73:7–15
32. Shahgholi H, Barani K, Yaghobi M (2017) Application of perfect mixing model for simulation of vertical roller mills. J Min Environ 8(4):545–553
33. Ahmed MM (2010) Effect of comminution on particle shape and surface roughness and their relation to flotation process. Int J Min Process 94(3–4):180–191
34. Pourghahramani P, Forssberg E (2005) Review of applied particle shape descriptors and produced particle shapes in grinding environments. Part I: particle shape descriptors. Mineral Processing & Extractive Metall Rev 26(2):145–166
35. Scott G, Bradshaw SM, Eksteen JJ (2008) The effect of microwave pretreatment on the liberation of a copper carbonatite ore after milling. Int J Min Process 85(4):121–128
36. Schmidt DC, Berg JC (1996) The effect of particle shape on the flotation of toner particles. Progress in paper Recycling 5:67–77
37. Schmidt DC, Berg JC (1997) A preliminary hydrodynamic analysis of the flotation of disk-shaped toner particles. Progress in Paper Recycling 6:38–49
38. Burstein MA, Hall ST, Kornyshev AA (1995) A study of the effect of particle fractal geometry on flotation performance, Proc Int Miner Process Congr Soc Min Metall Explor Littleton, Colorado, USA, vol. 3, pp. 77–80
39. Verrelli DI, Bruckard WJ, Koh PTL, Schwarz MP, Follink B (2014) Particle shape effects in flotation. Part 1: microscale experimental observations q. Min Eng 58:80–89
40. Anfruns JF (1977) Rate of capture of small particles in flotation, Transactions of the Institution of Mining and Metallurgy. Section C: Mineral Processing and Extractive Metallurgy 86:9–15
41. Yekeler M, Ulusoy U, Hiçyilmaz C (2004) Effect of particle shape and roughness of talc mineral ground by different mills on the wettability and floatability. Powder Technology 140(1–2):68–78
42. Kursun H, Ulusoy U (2006) Influence of shape characteristics of talc mineral on the column flotation behavior. Int J Min Process 78(4):262–268