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Effect of microwave system location on floatability of chalcopyrite and pyrite in a copper ore processing circuit

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Abstract: The present work aims at investigating the effect of microwave local positions (*i.e.* before crushing (BC), after crushing (AC) and after milling (AM)) on microwave-assisted flotation of chalcopyrite and pyrite in a porphyry copper complex deposit. Individual given samples for each state were pre-treated with a variable power microwave at a power level of 90 to 900W for 15, 30, and 60s. Furthermore, froth floatation experiments were carried out using a laboratory mechanical Denver flotation cell on both microwave-treated and untreated samples. Particle surface properties were characterized by a scanning electron microscopy (SEM) and an energy-dispersive X-ray spectroscopy (EDX) analysis. The results showed that the chalcopyrite and pyrite floatabilities increased monotonically by rising the exposure time and power level for the uncrushed preconditioned samples (BC) due to the enhancement of mineral liberation degrees together with the formation of sulphide species and polysulphides on the mineral surfaces. However, flotation results of treated samples for the crushed one (AC) revealed an optimum range. Formation of intensive oxide layers on the mineral surfaces of milled samples (AM) led to a substantial reduction in their recoveries by increasing the microwave's power level and the sample's exposure time. The results obtained from mineral's floatabilities in recleaner stage showed that the microwave-assisted sample at 900W for 30s at BC state favourably provided 5% higher S.E.'s than that of the untreated sample. Finally, it was concluded that the microwave pretreatment of samples induced the best floatability responses if it located before the crusher.

Keywords: microwave technology, chalcopyrite, pyrite, comminution circuit, separation efficiency

1. Introduction

Application of microwave (MW) to mining and mineral processing industries was studied from the early 1970s (Amankwah and Ofori-Sarpong, 2011; Andriese et al., 2011). Researchers initially employed it to facilitate the grinding and breakage processes concerning the enormous energy consumption of these operating units (Kingman et al., 2004; Andriese et al., 2011; Batchelor et al., 2016; Hassanzadeh 2017a; Batchelor et al., 2017; Gholami et al., 2019a). A substantial improvement in mineral liberation degrees through the microwave treatment in comminution stages are broadly reported in the literature. However, its influence on downstream units such as flotation has not been adequately addressed yet. Also, microwave irradiation of sulfide minerals (e.g. chalcopyrite and pyrite) under various exposure times and power levels are not profoundly comprehended and still fundamental concepts are required for clear understandings. Its scale-up challenges, modeling aspects and economic barriers are under

argument from different perspectives. Table 1 briefly presents the recent research works implemented on floatability of different types of materials pre-treated by the microwave highlighting their key findings.

Table 1. A short summary of the reported studies in the literature regarding the impact of microwave in flotation of different species highlighting their key conclusions

Author(s)	Mineral/Material	Notes
Kingman <i>et al.</i> , 1999	Norwegian ilmenite ore	Microwave radiation showed a significant effect upon the mineralogy and magnetic processing at shorter exposure times. Microwave treatment (1.3 kW) considerably increased the grade and recovery of the ilmenite in 10-20 s.
Kingman <i>et al.</i> , 2000	Palabora copper ore	Small initial increases in the recovery of copper were reported after microwave treatment. However, with prolonged exposure, recovery decreased.
Fan and Rowson, 2000	Norwegian high-grade massive ilmenite sample	Micro-flotation (using Hallimond tube) and rougher batch flotation (using Denver cell) experiments on MW-induced samples (2600W) indicated an enhancement of 24% on ilmenite's recovery.
Vorster <i>et al.</i> , 2001	Neves Corvo copper ore	Flotation trials on a microwaved sample and non-treated one showed insignificant improvement on copper grade and recovery.
Sahyoun <i>et al.</i> , 2005	South African copper carbonatite ore	Comparative batch flotation tests on MW-treated (at 5–12 kW for 0.1–0.5 s) and untreated samples revealed improvements in copper recovery of between 6–15 %. Both recovery and cumulative grade increased with increasing treatment time and power.
Muzenda and Afolabi, 2010	Low-grade nickel sulphide ore	The effect of oxygen, conventional air and microwave cracking was investigated. The amphibole mineral had the highest counts of all the minerals recovered. It was because of the activation of the ore using the microwave.
Batchelor <i>et al.</i> , 2016	Chilean porphyry copper ore	An increase in copper recovery of approximately 1% was reported. Preferential association of copper minerals with a hard matrix mineral (quartz) and a hard microwave-absorbent mineral (pyrite) resulted in a significant change in liberation behaviour.
da Silva <i>et al.</i> , 2018	Chalcopyrite sample	Surface properties of the microwave-treated and non-treated samples were characterized via XRD, XPS, ATR-FTIR, SEM, BET/BJH, IGC and ELS. Short exposure times were found to favor the formation of copper polysulphides at the chalcopyrite's surface, and longer treatments converted CuFeS_2 into iron oxides/hydroxides/oxyhydroxides and sulfate at the surface. Chalcopyrite's surface became energetically more active after longer treatment times, and the flotation recovery decreased.
Azghdi, and Barani, 2018	Chalcopyrite sample	Atomic force microscopy (AFM) analyses showed a decrease in the surface roughness of the chalcopyrite after microwave radiation owing to the oxidation of the surface and formation of iron oxides.
da Silva and Waters, 2019	Chalcopyrite, pentlandite and pyrrhotite samples	The effects of the exposure time on each mineral's recovery at different concentrations, pHs and particle sizes together with adoption measurements were studied through micro-flotation and electrophoretic zeta potential measurements. The floatabilities of studied minerals were affected differently by the microwave irradiation. Formation of non-uniform oxidation or patched layers after MW-treatment was proposed as the main reason for reducing recovery of chalcopyrite and pyrrhotite.

Generally speaking, each mineral has a specific heat capacity which causes differentiation in heating rate while being exposed to the microwave radiation. Some absorb heat more quickly while others react less to heat or not at all (Chen et al., 1984; Walkiewicz et al., 1988; Kingman et al., 2004). These behaviors help mineral processing engineers to separate valuable minerals selectively from gangue ones.

As widely known, chalcopyrite (CuFeS_2) is the world's most critical copper-bearing mineral which is often associated with pyrite (FeS_2) (Yianatos et al., 2014; Azizi et al., 2015; Agheli et al., 2018). Numerous studies are undertaken to investigate the effect of microwave treatment on their floatabilities. Variation of particle surface properties (i.e. roughness and hydrophobicity) as one of the crucial parameters under microwave irradiation is reported in several works (Table 1). The studies showed that the MW treatment affects the flotation behaviour of copper sulfide ores by a phase change in the boundary of chalcopyrite with gangue minerals during radiation (Marion et al., 2016). The absorbed microwave energy converts to heat in the material which may cause oxidation and alteration of mineral surfaces inducing a hydrophilic characteristic (Kaya, 2010; Gholami et al., 2019b). Many researchers have reported that the thermal pre-treatment can undermine the grain boundaries. It reduces the slime production in subsequent processes, increases particle's grindability and upgrade liberation of valuable minerals, together with conserving corrosion of equipment (Walkiewicz et al., 1988; King and Schneider, 1988; Veasey and Wills, 1991; Vorster, 2001; Walkiewicz et al., 2001).

Despite a wide number of research works dedicated to the role of microwave technology in grinding and flotation processes, there is a considerable lack of study on its location in processing flowsheets. The aim of the present investigation is to determine the right location of microwave pre-treatment in a typical processing flowsheet with respect to floatability of chalcopyrite and pyrite in a copper complex ore. With this purpose, microwave's local position in the circuit is studied through three states i.e. i) before crushing (BC), ii) after crushing (AC) and before milling (BM). Particle surface properties together with selective and effective separation efficiencies are discussed in detail for each case.

2. Materials and methods

2.1. Materials

2.1.1. Sample and its preparations

In this work, the initial samples (*ca.* 500kg) were taken from the feed of Sarcheshmeh copper concentration plant ($d_{80}=15\text{mm}$) situated in Rafsanjan, Iran. Detailed information concerning the ore body and the beneficiation plant can be found elsewhere (Hassanzadeh, 2015, 2017b, 2018). These samples were homogenized and subsequently split into three parts using cones and riffles. Each part was treated separately in terms of being exposed to microwave radiation, as shown in Fig. 1 and described as follows:

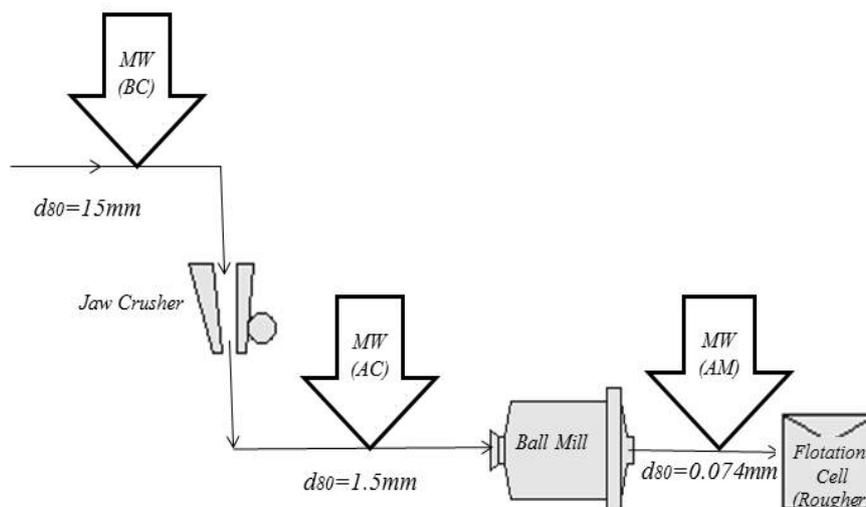


Fig. 1. Laboratory flowsheet designed for the ore treatment

I) Before crushing (BC); at first, 15 specimens containing 1kg representative samples ($d_{80}=15\text{mm}$) were taken from the first part and subjected to the microwave radiation under various time and power levels. After that, each specimen was crushed and ground by a jaw crusher (Retsch, BB100, Germany) and subsequently milled via a laboratory ball mill (standard Bond ball mill, England) to achieve $d_{80}=0.074\text{mm}$, specified for the flotation tests.

II) After crushing (AC); with respect to the second part of the samples, the selected representative specimens were divided into 1kg packages (with $d_{80}=1.5\text{mm}$) and exposed to microwave radiation. Similar to the first step and with the same manner, each sample was ground to reach $d_{80}=0.074\text{mm}$.

III) After milling (AM), like the previous two steps, the third part of samples ($d_{80}=0.074\text{mm}$) were crushed and ground under $74\mu\text{m}$. Then, they were packed in 1kg packages and each pack was treated with the microwave.

Eventually, untreated samples, along with all exposed specimens obtained from the three parts were used in the flotation experiments. The particle size distribution (PSD) of all three samples *i.e.* BC, AC and AM are displayed in Fig. 2. It is worth mentioning that the MW treatment naturally enhanced the mineral grindability which was dependent on the microwave's location leading to a variant product PSDs at the milling stage, prior to the flotation tests. Thus, to keep the PSD's effectiveness on the flotation experiments constant, the grinding time was selected in a way to reach $d_{80}=74\mu\text{m}$ for all the three states.

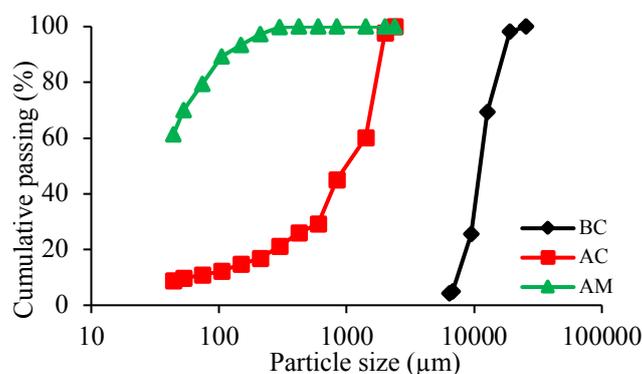


Fig. 2. The particle size distribution of the materials obtained from three stages of before crushing (BC), after crushing (AC) and after milling (AM)

2.1.2. Flotation reagents

The reagent types and dosages were selected according to the standard conditions of Sarcheshme copper processing plant. The mixture of sodium isopropyl xanthate ($\text{C}_4\text{H}_7\text{NaOS}_2$, Z11, 15 g/t) and butyl sodium dithiophosphate (Flomin 7240, 25 g/t) was used as collectors while the mixture of methyl isobutyl carbinol ($(\text{CH}_3)_2\text{CHCH}_2\text{CH}(\text{OH})\text{CH}_3$, MIBC, 102.174 g/mol, 15 g/t) and polypropylene glycol ($[\text{H}[\text{OCH}(\text{CH}_3)\text{CH}_2]_n\text{OH}$, F742, 134.17g/mol, 15 g/t) was employed as frother. More detailed information regarding the functionality of these chemical agents is reported elsewhere (Hassanzadeh *et al.*, 2019). 2 g lime (CaO) was added to the ball mill, and a sufficient amount of that was used to adjust the pH within the flotation experiments at 11.8.

2.2. Methods

2.2.1. Material characterizations

Chemical composition of the samples was characterized by an atomic absorption spectroscopy (AAS) (Varian, 220A) and X-ray fluorescence (Philips, 1480-PW model). Mineralogical analyses consisting identification of mineral mode and mineral associations were performed by spot counting method via optical microscopy and an in-house developed software at central laboratories of Sarcheshme copper complex. Table 2 presents the elemental and mineralogical compositions of the porphyry copper ore as a feed of the processing plant. According to the mineralogical analysis of the studied sample, the main precious copper mineral was chalcopyrite while the major minerals containing iron were chalcopyrite

and pyrite. Further, the predominant non-metallic gangue minerals were identified as quartz, illite, moscovite and albite.

To measure the liberation degree of individual minerals, the reflective optical microscopy (Leica-DMLSP, Germany) was used on the prepared polished sections. Scanning electron microscopy (SEM) (Phillips-XL30, The Netherland) equipped with energy dispersive X-ray analyzer (EDX) (Phillips-XL30, The Netherland) was utilized for evaluating the textural and morphological features and also the chemical composition of minerals surfaces before and after the microwave pre-treatments.

Table 2. Chemical and mineralogical compositions (wt%) of the studied sample

Mineral		Content (wt%)	Mineral		Content (wt%)						
Chalcopyrite (CuFeS ₂)		1.67	Pyrite (FeS ₂)		7.02						
Sphalerite (Zn, Fe)S		0.05	Hematite (Fe ₂ O ₃)		0.09						
Non-metallic minerals		91.13	Oxide minerals		0.02						
Elemental composition (wt%)											
CuO	Fe ₂ O ₃	MoO	SiO ₂	Al ₂ O ₃	SO ₃	CaO	MgO	K ₂ O	TiO ₂	P ₂ O ₅	L.O.I
0.74	5.50	0.016	52.61	15.40	7.31	1.35	4.09	5.97	1.05	0.29	5.67

2.2.2. Microwave pretreatments

Microwave pretreatment of the specimens was carried out by means of a power adjustable domestic microwave oven (Bosch model BER634GS1L, 2.45GHz) with a maximum power of 900W, and cavity dimensions of 382×594×318 mm. The treatment was conducted at power levels of 90, 180, 360, 600 and 900W for radiation times of 0, 15, 30 and 60 seconds. The samples were placed inside the oven in a transparent Pyrex glass container and then centered in cavity for all the tests.

2.2.3. Flotation experiments

Rougher flotation tests were performed on both the untreated and microwave-treated samples using a mechanically agitated Denver flotation machine with an impeller speed of 1400rpm in a 4.2L cell. Initially, the pulp with 28% solids was entered to the cell, then the pH was adjusted at 11.8 with lime and conditioned for 3min. After this period, the collectors and frothers were added with corresponding conditioning times of 1 and 2min, respectively. At the beginning of aeration, cumulative timed concentrates were collected at the intervals of 1, 2, 3, 5, 8 and 12min. Eventually, these six concentrates and tailings were filtered, dried and assayed.

In addition to the rougher flotation experiments, the cleaner and re-cleaner flotation tests were performed to investigate the flotation behavior of the chalcopyrite and pyrite after the microwave pre-conditioned in the final concentrate. For this purpose, the uncrushed and crushed microwave treated samples were processed after exposure times of 30 and 60s as schematically shown in Fig. 3.

Firstly, the rougher concentrate of each comminution stage was reground to reach a product size of $d_{80}=40\mu\text{m}$. Thereafter, the product was poured into a 2L flotation cell for preparation of the cleaner experiment. At this stage, 30% of collector dosage used in the rougher stage was added and the slurry conditioned for 2min at a pH of 12. The airflow was turned on and the froth collection was started for 3 minutes. Eventually, the concentrate of cleaning stage was poured into a 1L vessel for the re-cleaner tests. The final concentrate was obtained by collecting the froth phase at the intervals of 1, 2 and 3min. Fig. 3 illustrates the process steps for obtaining the final concentrate.

2.2.4. Calculations

To compare the effect of microwave pretreatment in each stage of comminution, the recovery of chalcopyrite and pyrite were calculated using Equation 1 (Wills and Napier-Munn, 2006).

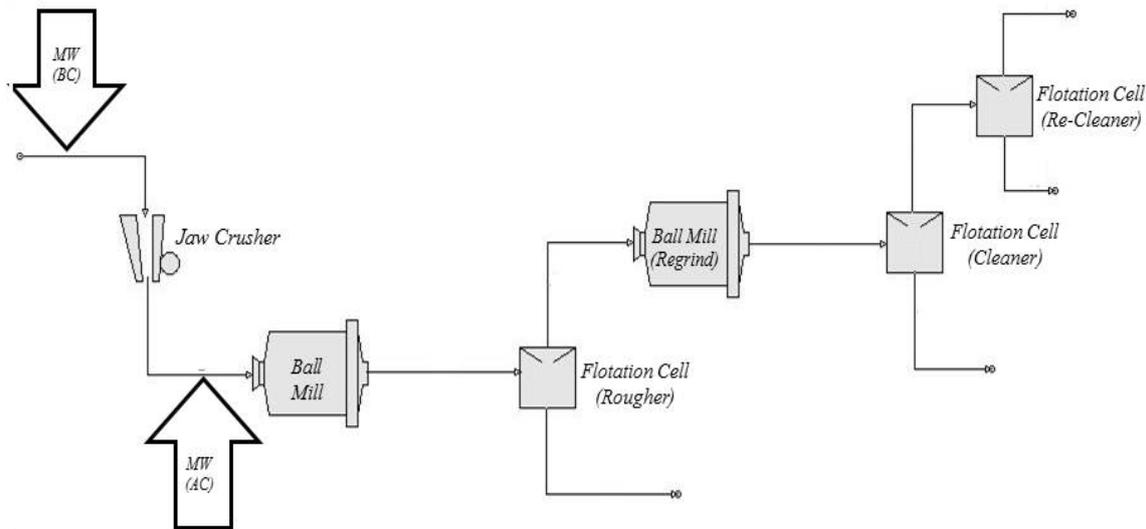


Fig. 3. A schematic view of the labrotary procedure representing the ultimate impact of the MW treatment on final concentrate

$$R = \frac{Cc}{Ff} \times 100 \quad (1)$$

where $R(\%)$ is recovery, C is the dry weight of the concentrate, c denotes the grade of concentrate and F and f represent the dry weight of feed and its grade, respectively.

In addition to grade and recovery, separation efficiency (S.E.) (Equation 2) was benefited as the key indicator for evaluating the metallurgical efficiency of the separation (Schulz, 1970; Wills and Napier-Munn, 2006):

$$S.E. = R_m - R_g = \frac{100Cm(c-f)}{(m-f)f} \quad (2)$$

where $R_m(\%)$ and $R_g(\%)$ are the recoveries of the valuable and gangue minerals in the concentrate, respectively. Assuming c , f and t are the metal content in the concentrate, feed and tailing, respectively. C is the fraction of the total feed weight that reports to the concentrate and m is the theoretical metal content in the valuable mineral.

3. Results and discussion

3.1. Microwave irradiation before crushing (BC)

Fig. 4 exhibits the effect of microwave irradiation in the sense of time-dependent prior to crushing stage on chalcopyrite and pyrite recoveries. It is explicitly seen that the recovery of both minerals is enhanced by increasing the irradiation power and exposure time. The recovery of chalcopyrite increases by approximately 5% while being exposed for 60 s at a power level of 900 W in comparison to the untreated sample. At these comparisons of irradiation time (at the same power level), the maximum flotation recovery of chalcopyrite is almost 88% under microwave pretreatment for 60 s at a power level of 900 W. The pyrite recovery also increases and reaches 36% which is almost 12% greater than the untreated pyrite. These findings are in good agreement with previous studies (Batchelor et al., 2016; Marion et al., 2016) demonstrating that the microwave pre-treatment improves the degree of particle liberation, and consequently affects the minerals floatability positively.

It is worth noting that the selective separation of chalcopyrite from pyrite typically occurs at alkaline pHs (>11) at rougher flotation stage where pyrite underwent extreme depression (Gaudin, 1939; Hassanzadeh and Karakas, 2017). However, as widely reported in the literature (Shen et al., 2001; Agorhom et al., 2015; Hassanzadeh and Hassanzadeh, 2017) and the observed results, despite an increase at pH value (i.e. pH>11), pyrite particles show good affinity to flotation. It can be mainly contributed to the positive effect of xanthate concentration, natural hydrophobicity of pyrite in fresh surfaces, surface reactions and electrochemical processes (Weisener and Gerson, 2000; Mermillod-Blondin et al., 2005).

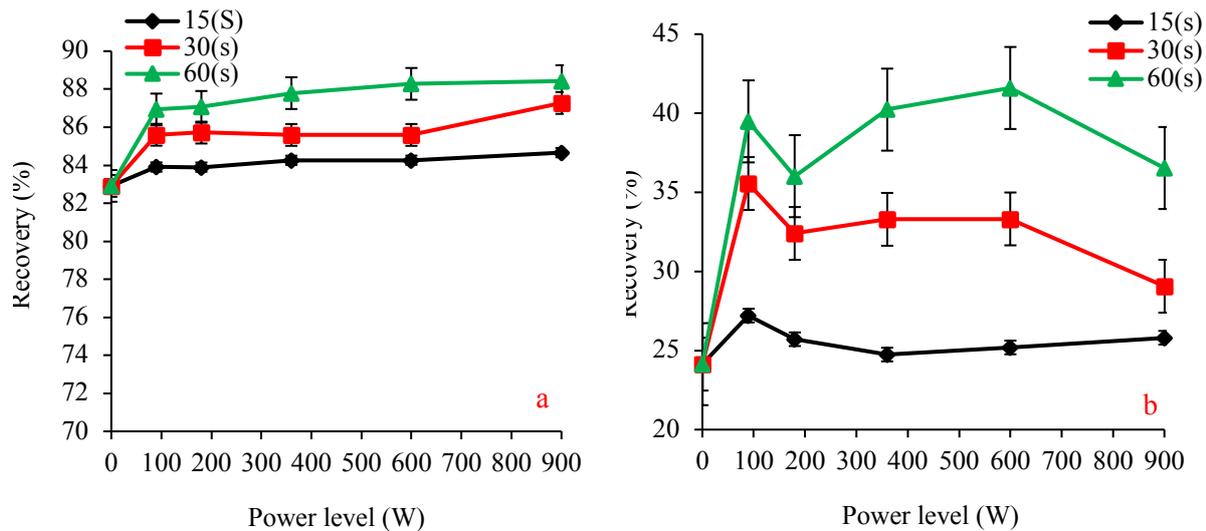


Fig. 4. The variation of a) chalcopyrite and b) pyrite recoveries as a function of power and exposure time of microwave treatment

3.2. Microwave irradiation after crushing (AC)

The floatability outcomes of the ore treatment after crushing (prior to milling stage) are illustrated in Fig 5. As seen, at a constant microwave power, the maximum chalcopyrite and pyrite recoveries occurred after irradiation for 30 s. The more increase in exposure time decreases both minerals' recoveries dramatically. The highest improvement in the flotation recovery of chalcopyrite is taken place after radiation at a power level of 900W for 30s. Under these conditions of radiation, chalcopyrite and pyrite recoveries are 88% and 34%, respectively. At a radiation power of 900 W, when the exposure time increases to 60 s, their flotation recoveries reach 80.5% and 29.5%, respectively. These results evidence that at a constant power of microwave irradiation by reducing material's size from $d_{80}=15$ mm to $d_{80}=1.5$ mm, the optimal exposure time diminishes from 60 s to 30 s.

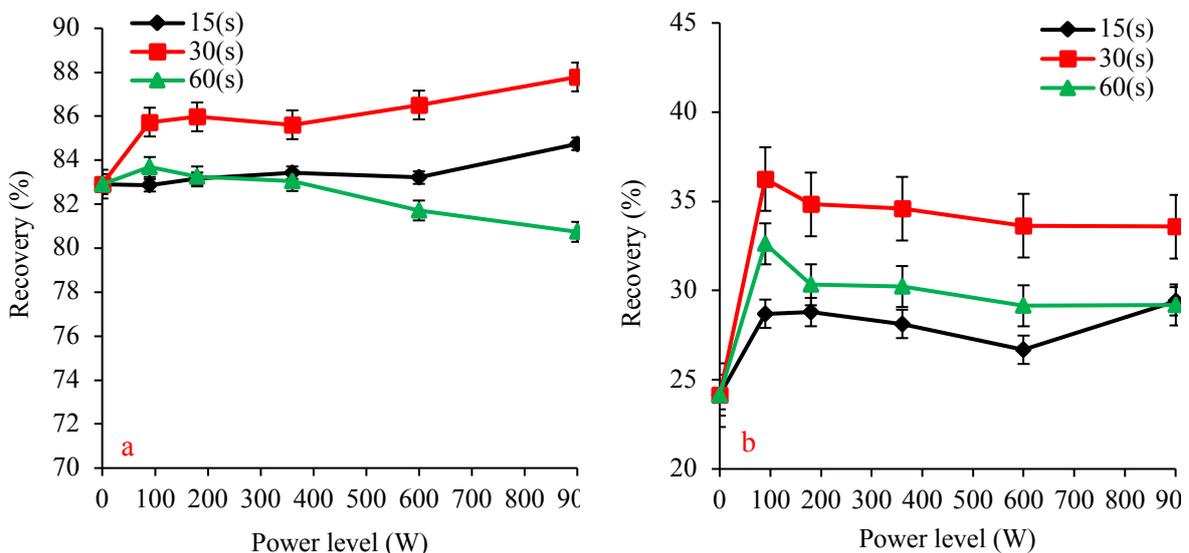


Fig. 5. The variation of a) chalcopyrite and b) pyrite recoveries as a function of power and exposure time of microwave irradiation

3.3. Microwave irradiation after milling (AM)

Fig. 6 exhibits the chalcopyrite and pyrite recoveries for untreated and microwave-treated samples for AM ($d_{80}=0.074$ mm). As shown in Fig. 6, ore treatment prior to flotation stage affects the flotation

recoveries of chalcopyrite and pyrite negatively. Unlike two prior states *viz.* BC and AC, in this stage of comminution in which more than 80% of chalcopyrite and pyrite have been liberated, the flotation recoveries of both minerals are decreased by increasing the microwave exposure time and power level. In the case of chalcopyrite, when the microwave power reaches 900 W the most reduction in recoveries takes place in all three exposure times. At the third exposure time, despite the increase in microwave power, the floatability of chalcopyrite remains constant. In other words at high powers, the flotation recovery of chalcopyrite depends on the microwave radiation time. More the exposure time, lower the floatability of chalcopyrite. For instance, chalcopyrite's recovery drops from 83% for untreated sample to approximately 75% and stays constant at 74% despite the power level increases (Fig. 6a). For sample treated under 900 W for 60 s, the pyrite's recovery monotonically diminishes by increasing either power level or irradiation time. As can be explicitly seen in Fig. 6b, pyrite flotation recovery in the sample pretreated by 900 W microwave at 60 s shows the lowest value.

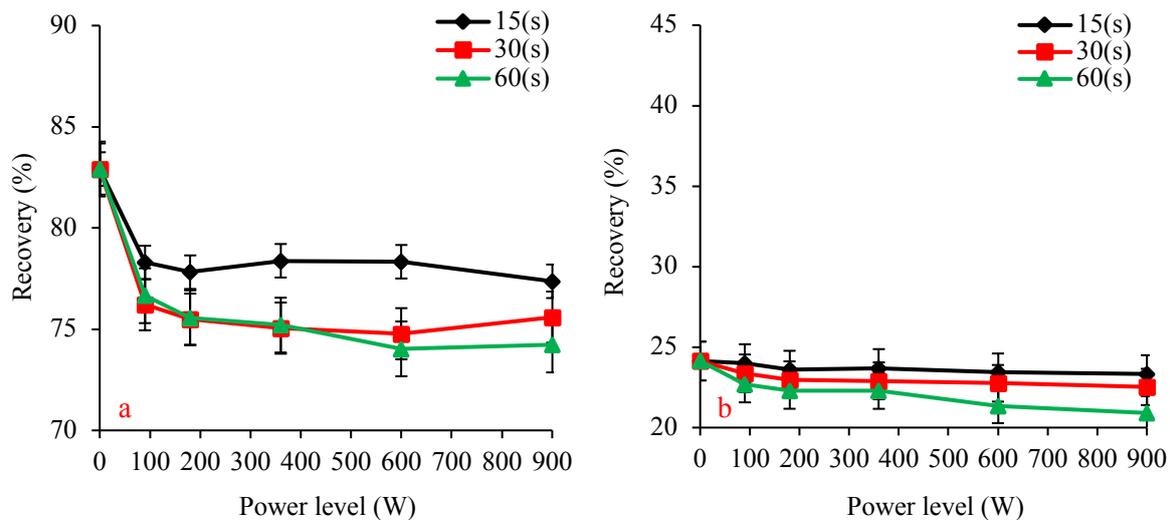


Fig. 6. The variation of a) chalcopyrite and b) pyrite flotation recoveries as a function of power and exposure time of microwave irradiation

According to the obtained results, it can be concluded that microwave technology leads to an improvement of overall chalcopyrite recovery for exposure times of 60 and 30 s for uncrushed and crushed samples, respectively. Whereas, at all the radiation durations, pyrite's recovery enhances in the case of uncrushed and crushed samples compared to the untreated state. Therefore, treated samples for 30 and 60 s at 900 W microwave power are chosen to simplify the analyses of particle liberation degree for uncrushed and crushed samples.

3.4. Liberation degree of minerals

To investigate the effect of microwave irradiation at various stages of comminution on flotation behavior of chalcopyrite and pyrite, the liberation degree (LD) of these minerals was determined after grinding the different samples under 74 μ m before and after pretreatment. To estimate the LD, the microscopic studies were carried out on the un-microwaved and microwaved samples before and after the crushing. The results are illustrated in Fig. 7 typically categorizing the mineral liberation to liberated (LD>90), middling (30<LD<90) and locked (LD<30) particles which are calculated on the basis of weight percentage using Gaudin's liberation model (Gaudin, 1939).

As shown in Fig. 7, by increasing the exposure time from 0 s to 60 s, the amount of fully liberated chalcopyrite and pyrite particles increases. It can be also seen that at the same exposure time, the amount of liberated particles in the treated crushed (AC) sample is reasonably greater than that of the irradiated non-crushed (BC) sample. In comparison with untreated sample, the liberation degree of chalcopyrite in the AC and BC samples exposed to microwave for 60 s increases 33% and 9%, respectively. With regard to Fig. 7b, the LD of pyrite reaches 92% and 70% for crushed and non-crushed samples, respectively which shows an increase of almost 32% and 10% in comparing with un-treated one. The

increase of LDs can be attributed to the differences in heat absorption rates of various minerals leading to the creation of micro-fractures and micro-cracks between the grain boundaries (Walkiewicz et al., 2001; Kingman et al., 2004; Svensson et al., 2017).

By combining the results obtained from bulk flotation experiments (Figs. 4-5) and mineralogical and microscopic analyses (Fig. 7), one can conclude that solely by rising the exposure time from 0 to 30 s for the AC samples, chalcopyrite and pyrite recoveries improve. When the exposure time is 60 s, the recoveries and flotation rate constants significantly drop down owing to the formation of oxide species on the surface of the mineral.

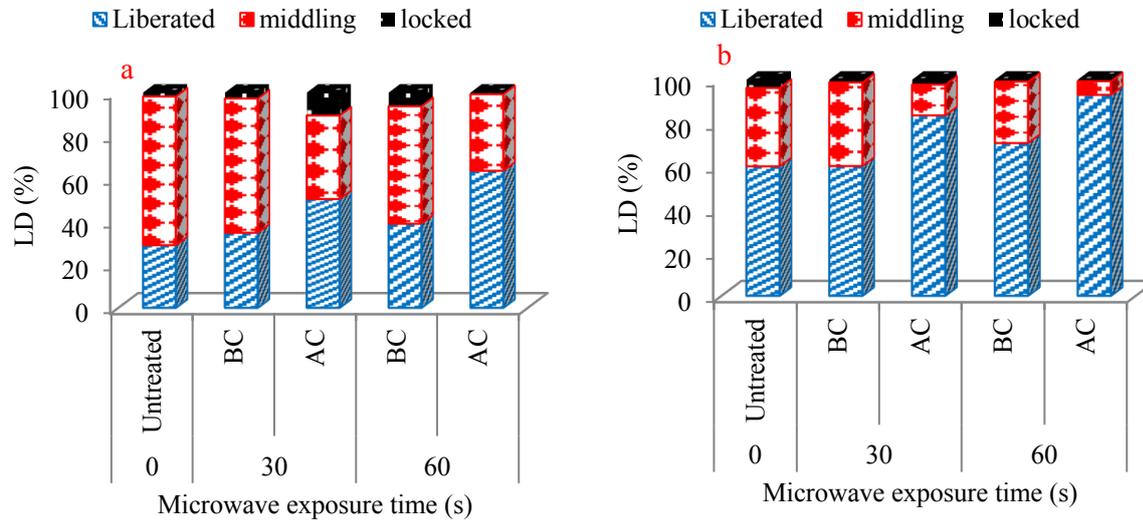


Fig. 7. The LDs of a) chalcopyrite and b) pyrite in the untreated and microwave treated samples (irradiation time: 0, 30 and 60 s)

3.5. Scanning electron microscopy (SEM) studies

For investigating the variation of mineral floatability via microwave pretreatment, the surface of treated and untreated chalcopyrite and pyrite was studied by SEM equipped with EDX from morphological and chemical compositional points of view. The results are illustrated in Figs. 8 and 9. The SEM images are captured from the surface of liberated chalcopyrite and pyrite particles with the size of $-74\mu\text{m}$ (produced AM). These results show that the surface of the untreated particles of chalcopyrite and pyrite is smooth and uniform (Figs. 8a and 9a) while it is altered to non-smooth surfaces after microwave irradiation at different stages of comminution. Fig. 8b shows the surface of the liberated chalcopyrite after microwave irradiation of uncrushed ore sample. As seen in Fig. 8b, some light particles have been appeared on the mineral surfaces, and the roughness of the chalcopyrite surface has been increased. As shown in Figs. 8c and 8d, the appearance of light particles on the surface of chalcopyrite are enhanced when the crushed (AC) and milled (AM) ore samples have pretreated by microwave radiation. In the case of milled ore sample, some pores are also created on the surface of chalcopyrite through microwave pretreatment (Nuri et al., 2014; Mehdilo and Irannajad, 2016). Indeed, heat adsorption might cause phase transformation and formation of different oxidation layers which starts from the surface of the mineral and continues to the core of each particle (Can and Bayraktar, 2007).

In addition to the SEM images, the chemical compositions of the chalcopyrite and pyrite surfaces are displayed by EDX spectra for untreated and microwave-treated samples. The summarized results given in Fig. 10 show that in the case of un-crushed samples (Fig. 8a, b and 9a, b), microwave impact on the surface oxidation of mineral is somewhat mild. The oxygen portion on the surface of chalcopyrite and pyrite is increased correspondingly from 6.32% to 10.87% and 5.94% to 7.52% for the uncrushed sample (BC). For this sample, the copper content on the surface of chalcopyrite decreases slightly from 22.13% to 19.15%. On the other side, the sulfur content in the surface of chalcopyrite enhances from 45.58% in untreated sample to 47.44% in BC sample after microwave irradiation, which is approximately 60% in the first stage of pyrite's treatment implying the formation of sulphide species on particle surfaces.

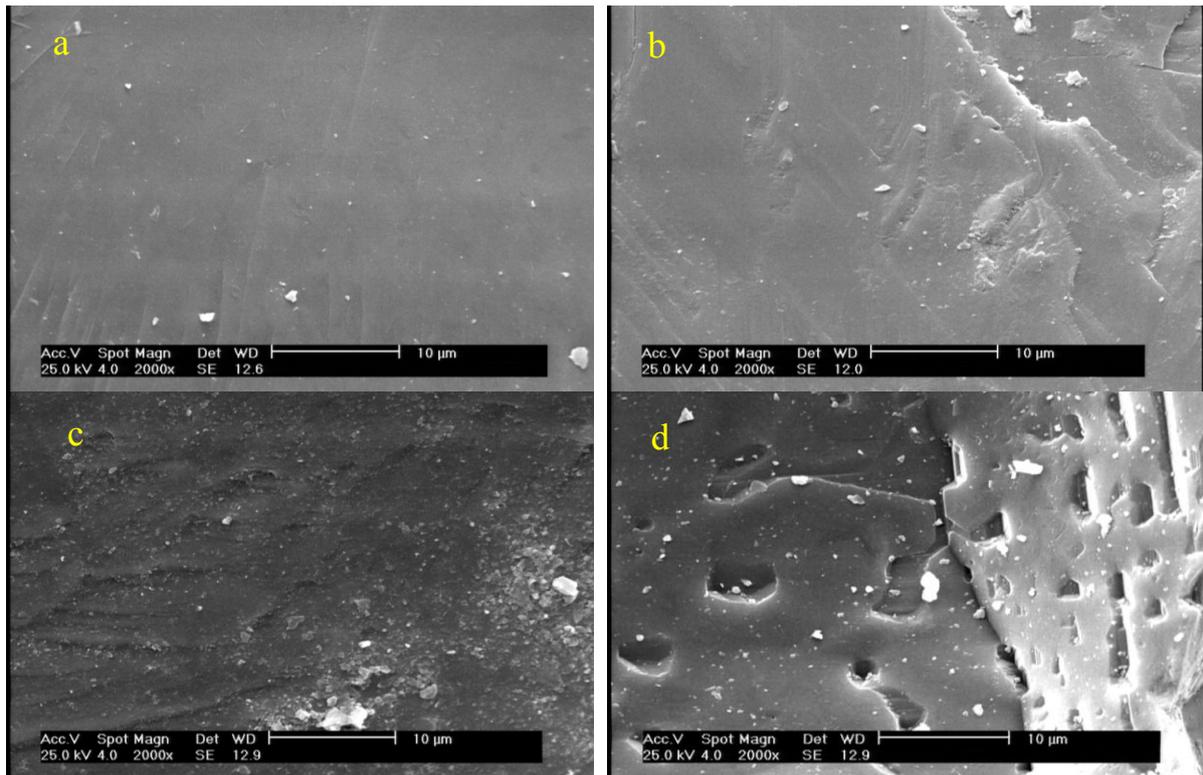


Fig. 8. SEM images of untreated and microwave treated chalcopyrite surface at various stage of comminution for a) untreated b) un-crushed, c) crushed and d) milled samples (900 W, 60 s)

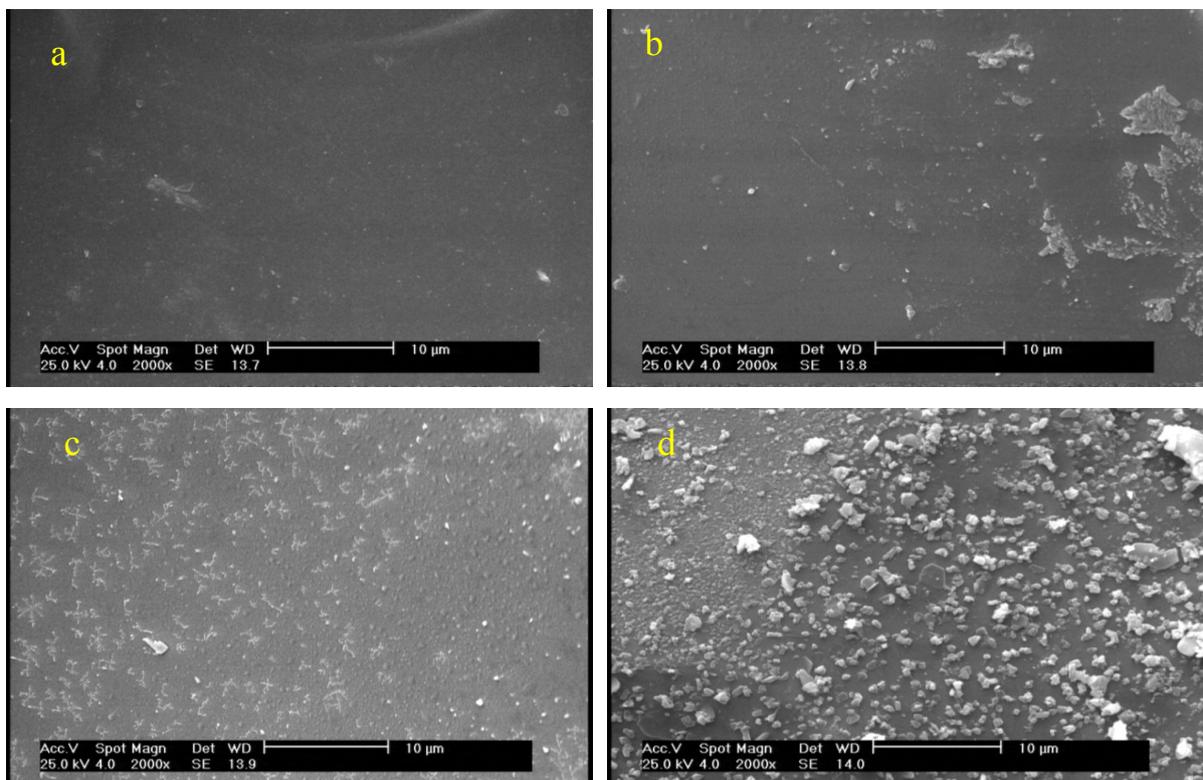


Fig. 9. SEM images of untreated and microwave treated pyrite surface at various stage of comminution for a) untreated b) un-crushed, c) crushed, and d) milled samples (900 W, 60 s)

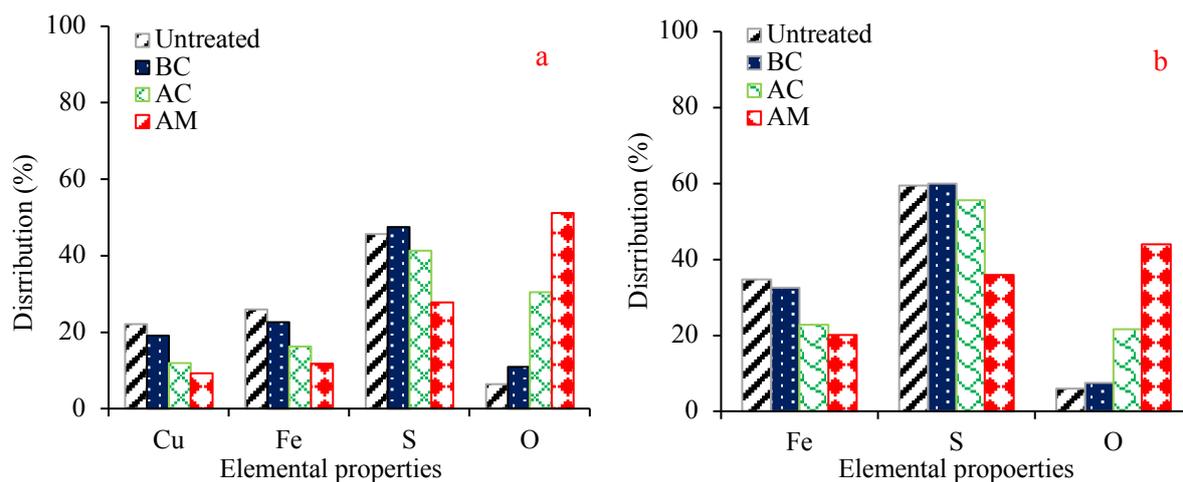


Fig. 10. Chemical analysis of chalcopyrite and pyrite before and after microwave irradiation by EDX analyzer (900W, 60s) radiation applied to the product of BC, AC and AM

It is worth noting that the superficial oxidation of some species with respect to the untreated samples naturally taking place because of the rapid reaction between sulphide surfaces with water and oxygen in the medium (da Silva and Waters, 2018). This slight oxidation unavoidably occurs during crushing and milling processes.

As shown in Fig. 8 and Fig. 9 (c and d) in the AC and AM samples, microwave irradiation impact on the variation of particle surface characteristics increases by reducing the average particle size. For instance, the oxygen content on the surface of chalcopyrite belonging increases from 6.32% in the untreated sample to 30.42% and 51.2%, respectively. In the case of pyrite, it rises up from 5.94% to 21.58% and 44.44% for the crushed (AC) and milled (AM) samples, respectively.

It is widely known that minerals have different thermal absorption rates which are related to their dielectric constants (Kingmann et al., 2000). The heat absorptions rate is high for both chalcopyrite and pyrite although pyrite is more resistant than chalcopyrite against microwave radiation. Thus, its temperature is lower than chalcopyrite (McGill et al., 1988) while being subjected to a constant period of exposure time. This is a possible reason causing a reduction on oxide species on pyrite's surface compared to that on chalcopyrite (Elmahdy et al., 2016). This can be explicitly seen for all three samples by consideration of oxygen component for chalcopyrite and pyrite resulting in intensive oxidation of chalcopyrite's surface than pyrite's one. The consequence can be related to the reduction of chalcopyrite's recovery in the AC sample irradiated in comparison with the untreated sample under exposure time of 60s (Fig. 5).

According to the results obtained from the overall flotation experiments, liberation studies, and surface properties of particles, it can be concluded that the local position of microwave prior to crushing (BC) and after crushing (AC) improves the floatability of chalcopyrite and pyrite while their flotation recoveries are reduced when the irradiation takes place after milling (AM sample). Therefore, BC and AC were selected to evaluate the metallurgical process.

3.6. Metallurgical evaluation

In addition to the rougher flotation stage, the cleaner and re-cleaner tests were performed as demonstrated in Fig. 3. Fig. 11 displays the chalcopyrite and pyrite resultant grades and recoveries in the final concentrate (re-cleaner stage) for the untreated and the microwave-treated states at radiation periods of 30 s and 60 s. As shown in Fig. 11a, the grade and recovery of chalcopyrite are increased after treatment at BC and AC states and only decreases for AC sample after an exposure time of 60 s. The maximum grade and recovery in the final concentrate of chalcopyrite is related to the exposure time of 30 s for BC sample. In the case of pyrite (Fig. 11b), maximum and the minimum grades and recoveries are obtained at the exposure time of 60 s for BC and AC states, respectively.

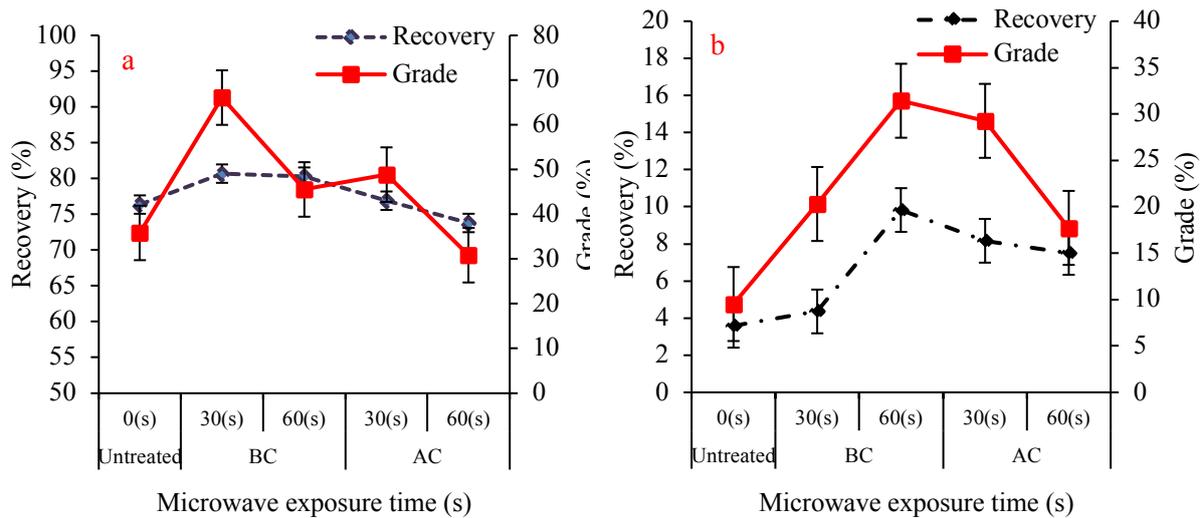


Fig. 11. Grade and recovery of a) chalcopyrite and b) pyrite in the final concentrate for the untreated and MW-treated samples of BC and AC as a function of exposure time at 900 W

Since the aim of the copper processing circuit is to increase the floatability of chalcopyrite while depressing pyrite, their separation efficiencies (S.E.s.) are calculated using Equation 2 for either the untreated or MW-treated samples at BC and AC modes.

Fig. 12 illustrates the S.E. values for the minerals of interest at the re-cleaner stage for the untreated and microwave-treated samples of the uncrushed and crushed modes at 30 and 60 s. As seen, the selective separation for the chalcopyrite is reasonably favorable if the uncrushed samples (BC) are pre-treated with microwave. The S.E. value of BC sample excited for 30 s is almost 80%, while it drops down to 71% for AC sample at an exposure time of 60 s. In the case of pyrite, the trend reaches the highest level for BC sample at the irradiation time of 60 s while the S.E. value for the sample AC plunges with increasing the radiation time. Unlike chalcopyrite, pyrite's S.E. values under any microwave treatment condition are always greater than that of the untreated ones.

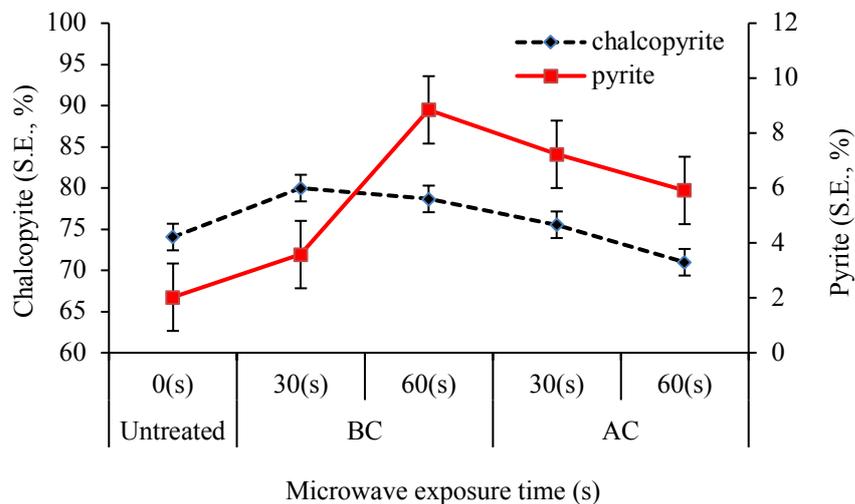


Fig. 12. The S.E. values calculated for the chalcopyrite and pyrite at the final re-cleaner stage for the untreated and MW pre-treated samples at 900W

4. Discussion

According to the results, microwave treatment has a different role in changing liberation degree and physicochemical properties of the minerals at various stages of the pretreatment. In BC, increasing the exposure time of microwave leads to improving the LD and alteration of the chemical compositions of

minerals' surfaces compared to the untreated state. According to the results of the EDX, a slight increase in the oxygen and sulfur magnitudes indicates poor oxidation on the particle surfaces. According to reported studies (Hayes and Ralston, 1988; Ghahremaninezhad et al., 2010; Can, 2016; da Silva et al., 2018), in cases of mild oxidation of chalcopyrite and pyrite, polysulphides and metal deficient sulphides are formed on the surface of the particles leading to hydrophobization and stability of the surfaces. Consequently, it increases the recovery of sulfide minerals as previously shown in Fig. 4. Therefore, rising the irradiation exposure time from 0 s to 60 s induces an improvement on the particle LD and in turn alteration of chemical compositions on the mineral surfaces.

In terms of AC, with the size reduction of treated particles, the effect of microwave irradiation on the liberation and changes of surface properties of particles is greater than the BC. In this context, Walkiewicz and Chanaa (Chanaa et al., 1994; Walkiewicz et al., 1988) found that finer particles exhibited favorable thermal absorptions than the coarser particles. As noted earlier, although an improvement in the LD is taken place for AC, increasing exposure time from 30 s to 60 s diminishes floatability of the sulphid minerals (Fig. 5). Following this, studies by Orumwense and Negeri, 2004 on a sulphide ore containing pyrite, chalcopyrite, galena and sphalerite manifested that the copper recovery slowed down after exposing to microwave radiation. With respect to the results reported by Sahyoun et al., 2005, the copper recovery of carbonate ore increased significantly during short microwave radiation but they believed the recovery was susceptible to power levels and exposure time of microwave radiation.

With respect to the AM, by increasing the time and power of the microwave radiation, floatability of chalcopyrite and pyrite reduced. With this regard, Can and Bayraktar, 2007 observed that the floatability of pyrite and chalcopyrite were reduced with increasing the power and radiation time which is in absolute agreement with our findings. Kingman et al, 2000, also showed the same phenomenon for sulfide minerals which oxidized when exposed to rich oxygen, and phase changes occurred at their surfaces. These superficial changes can reduce the recovery of valuable particles (Sahyoun et al., 2003). More specifically, in these two cases viz. AC and AM, increasing oxygen proportion and reduction of sulfur's content compared to the untreated and un-crushed treated samples indicates formation of oxidation layers on chalcopyrite and pyrite surfaces. In a same manner, a significant increase in conjunction with oxygen content for the milled sample points out the presence of oxidative species at chalcopyrite and pyrite surfaces. As reported in the literature, these species are oxides, hydroxides and oxyhydroxides (da Silva et al., 2018; Mita, 2018) which reduce the recovery of sulfide minerals in the AC and AM (Fig. 5 and Fig. 6). In addition to increasing the oxygen, reduction of surface sulfur leads to the formation of sulfate (SO_4^{2-}) on the mineral's surface (Elmahdy et al., 2016). Klauber et al., 2008 believes that at this stage, the surface sulphur layer formed are crustily and as shown in Fig. 7d, porous layers have been created on the mineral surface. The amount of iron had been decreased in all three stages of pretreatment at the surface of two minerals that indicating the formation of moderate oxide layers (ferric sulfate ($\text{Fe}_2(\text{SO}_4)_3$)) (Elmahdy et al., 2016). Increasing the power level of the microwave leads to an intense oxidation and creates magnetic phases such as Goethite (FeOOH) and Hematite (Fe_2O_3) on the mineral surface. Kingman et al, 2000, showed that magnetic capabilities of a number of minerals such as chalcopyrite, hematite, and wolframite were improved after microwave radiation. This enhancement had also been observed in minerals like pyrite, arsenopyrite and tetrahedrite (Lovas et al., 2003; Znamenáčková et al., 2005; Farahat et al., 2017). In contrast, the recovery of ilmenite increased after exposing to the microwave radiation because surface oxidation increased the absorption of oleate on the surface of ilmenite (Fan and Rowson, 2000; Fan and Rowson, 2002).

Following the cases studies discussed above and results obtained, one can conclude that different results in the S.E. values might be attributed to the degree of liberation and surface oxidation of minerals. It can be seen that the S.E. values for chalcopyrite are always greater than those for pyrite. One possible reason is contributed to the kinetic rate differences between chalcopyrite and pyrite as well as their surface characteristics in terms of adsorption of reagents for covering hydrophobic layer on their surfaces (Hassanzadeh and Hassanzadeh, 2016).

5. Conclusions

The present study investigated the effect of microwave irradiation and its local position in a mineral processing plant on floatability and surface properties of chalcopyrite and pyrite in a porphyry copper

ore. With this purpose, microwave's local position was examined in three states *i.e.* before the jaw crusher (BC), after the jaw crusher (AC) and after the ball mill (AM). Bulk and kinetic rougher flotation experiments were carried out in a laboratory mechanical flotation cell. Further, SEM and EDX analyses were used for identification of any variation on particle surface properties.

The results obtained showed that the impact of microwave technology on floatability of chalcopyrite and pyrite minerals strongly depended on its location in the processing circuit. Un-crushed and crushed microwave-induced samples showed a significant improvement on particle liberation degrees and property changes in the mineral surfaces. However, microwave treatment of milled samples was limited to the surface and phase changes of the particles.

The results driven from the flotation experiments disclosed that the microwave's effect on the recovery of chalcopyrite and pyrite was respectively positive for un-crushed sample, positive and negative for crushed one, and negative for milled sample. In addition to increasing the liberation degree, the mild oxidation and the formation of sulphide species on the surface of chalcopyrite and pyrite promoted flotation kinetics. Moreover, formation of intensive oxidized phases on the milled chalcopyrite's surface caused a dramatic reduction on its floatability at the studied time intervals and power levels. Finally, the S.E. values revealed that the selection of microwave's local position before crushing could improve the technical performance of a concentration process. Therefore, proper local position of microwave in the circuit together with shorter exposure time and high power level of irradiation might eventually cover the economic concerns of microwave's application to mineral processing plants.

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