

# Effect of Ultrasonic Pre-Treatment and Aeration on Flotation Separation of Chalcopyrite from Pyrite

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**ABSTRACT:** *In this paper, the effect of ultrasonic pre-treatment and aeration on the flotation separation of chalcopyrite from pyrite was investigated at different amounts of potassium ethyl xanthate (KEX), as a collector, and pH values. Artificially-mixed samples of chalcopyrite and pyrite were subjected to flotation studies to understand the effect of aeration with or without ultrasonic treatment on the flotation behavior of the minerals. Results of laboratory micro-flotation tests indicated that joint aeration and ultrasonic treatment makes it possible to separate chalcopyrite from pyrite effectively. X-ray Photoelectron Spectroscopy (XPS) confirmed the existence of negligible amounts of hydroxide/oxide species, which are resulted from galvanic interactions, on the surfaces of the minerals after conditioning of the suspension by ultrasound waves. The effective separation of chalcopyrite from pyrite was attributed to desorption of metal hydroxide precipitates, as hydrophilic species, from the surface of the chalcopyrite by ultrasonic treatment.*

**KEYWORDS:** *Flotation; Separation; Chalcopyrite; Pyrite; X-ray photoelectron spectroscopy; Aeration; Ultrasonic pre-treatment.*

## INTRODUCTION

Pyrite as an iron sulfide mineral is often in association with chalcopyrite. Pyrite is commonly considered as a gangue in chalcopyrite flotation. It is well known [1-4] that, galvanic interaction between chalcopyrite and pyrite affects floatability of the minerals, making it difficult to effectively separate chalcopyrite from pyrite. Activation of pyrite by the Cu extracted from chalcopyrite and formation of hydroxide layer on chalcopyrite have been reported [5, 6] as the results of galvanic interactions. In such a galvanic cell, pyrite with higher rest potential

acts as a cathode while chalcopyrite with lower rest potential acts as an anode, and electrons flow from chalcopyrite to pyrite (Fig. 1) in which hydroxide species form on chalcopyrite. Thus, these phenomena result in an increase and decrease in floatability of pyrite and that of chalcopyrite, respectively.

In order to separate chalcopyrite from pyrite, it seems necessary to remove hydroxide/oxide species (resulted from the galvanic interaction) from the surface of chalcopyrite. Although different studies have investigated

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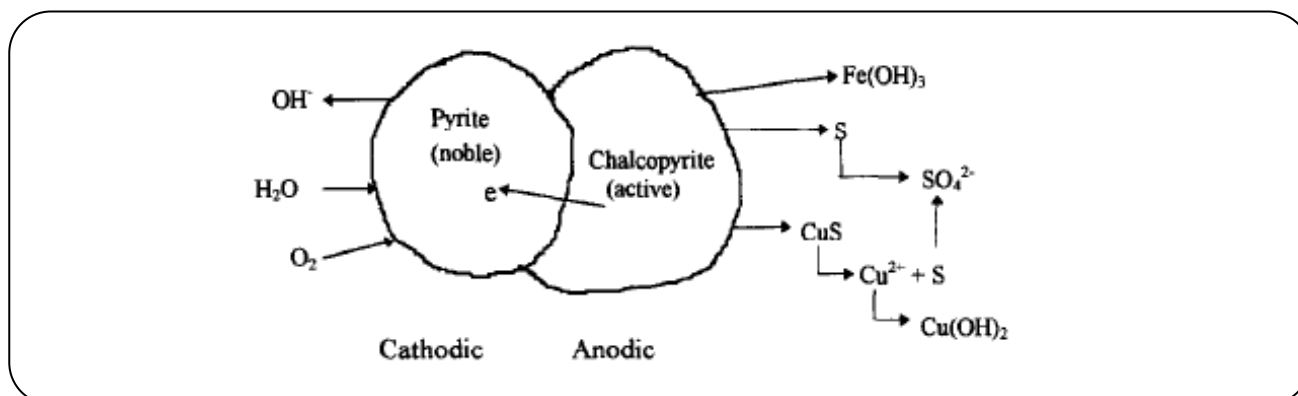


Fig. 1: Galvanic interaction between chalcopyrite and pyrite [2].

the problem, there is no significant study regarding surface cleaning effect of ultrasound waves when it comes to the removal of hydroxide/oxide precipitates from the surfaces of minerals.

The use of ultrasound to clean surfaces of particles in suspension is well known [7, 8]. Ultrasonic treatment has been also used as a preconditioning method to beneficiate minerals by flotation process [9-13]. When a suspension is ultrasonically treated, the waves travel through the liquid by periodically generating compression and rarefaction forces, releasing a very large and localised burst of energy. This phenomenon is known as cavitation. The energy released by cavitation can be utilized to clean surfaces of minerals suspended in liquid [7, 11, 14].

Therefore, the main objective of the current study was to investigate the surface cleaning effect of ultrasonic treatment on desorption of hydroxide/oxide species from the surface of the minerals, so as to improve the separation of chalcopyrite from pyrite.

## EXPERIMENTAL SECTION

### Materials and methods

Manually selected single crystals of chalcopyrite and pyrite were supplied from high-grade lump materials from Sarcheshmeh Copper Mine, southeastern Iran. The lumps were crushed to below 15 mm in a laboratory jaw crusher. They were then ground to below 100  $\mu\text{m}$  using a laboratory ceramic ball mill, from which particles of below 37  $\mu\text{m}$  in size were removed. Afterwards, the samples were packed in sealed polyethylene bags and stored in liquid nitrogen to prevent oxidation. The particle size of 75  $\mu\text{m}$  (i.e.  $d_{80}$ ) was measured for both samples. Elemental compositions of the samples, as obtained

by atomic absorption spectroscopy (AAS-Varian, A 220) and X-Ray Fluorescence (XRF-Philips, PW 1480), are given in Table 1. Prepared chalcopyrite and pyrite samples were of 97.37% and 98.18 % purities, respectively.

Potassium ethyl xanthate (KEX) and Dowfroth 250 were used as collector and frother, respectively. Hydrochloric acid (HCl) and sodium hydroxide (NaOH) were used to regulate pH of the suspension. All of the used chemicals were of analytical grade.

### Ultrasonic treatment

A probe ultrasonic transmitter of a total power of 700 W (with variable power levels) at a frequency of 20 kHz was used in the tests. A mixed sample of minerals containing 2.5 g of chalcopyrite and 2.5 g of pyrite was mixed with 100 mL of distilled water in a micro-flotation cell equipped with an air inlet valve and cooled by a tap water circulating system. Ultrasonic treatment of the suspension was performed for 15 min at 40% of total output power during the entire conditioning time. Fig. 2(a) shows the experimental setup schematically.

### Flotation tests

All flotation tests were carried out in the above-mentioned micro-flotation cell (Fig. 2(b)). In each test, 5 g of the sample was introduced into 100 mL of distilled water. In order to prepare a suspension of the mixed minerals, 2.5 g of chalcopyrite and 2.5 g of pyrite samples were added to 100 mL of distilled water. After adjusting pH of the suspension to desired values by dilute HCl or NaOH solutions, the suspension was conditioned via aeration for 15 min with or without ultrasonic treatment.

Table 1: Elemental compositions of the chalcopyrite and pyrite crystals.

Sample	Elements (wt%)							
	Cu	Fe	S	CaO	K <sub>2</sub> O	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	others
Chalcopyrite	34.16	30.08	34.46	0.02	0.05	0.36	0.22	0.65
Pyrite	0.28	30.08	52.39	-	-	0.56	0.68	0.27

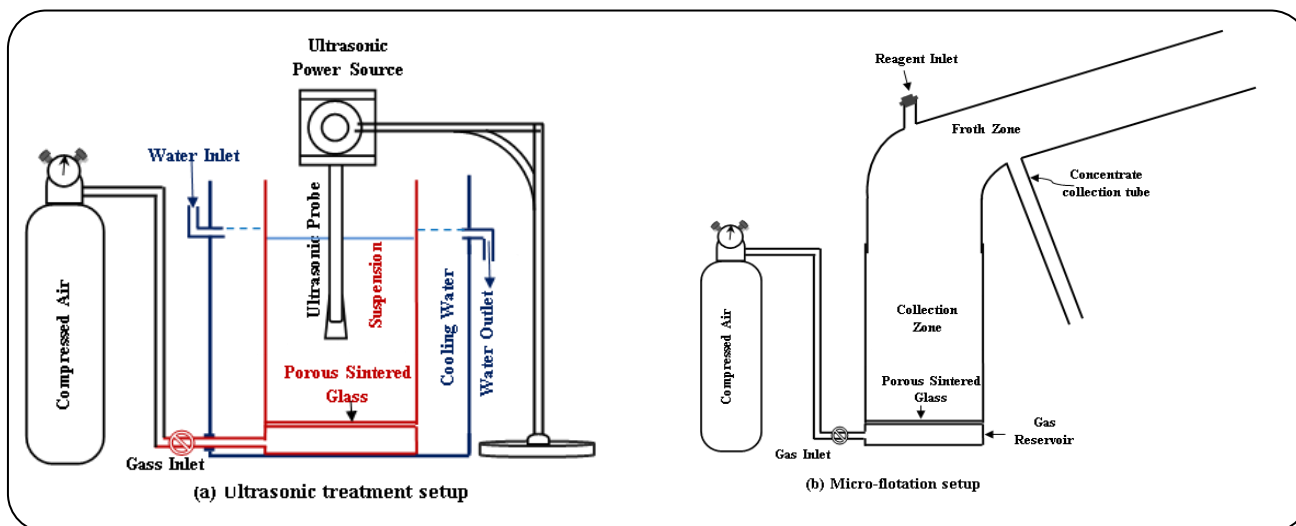


Fig. 2: Schematic diagram of the experimental (a) ultrasonic treatment (b) flotation setup.

The collector was introduced into the cell 3 min before the end of conditioning time. The flotation concentrates were then collected for 15 min after adding the desired amount of frother ( $1.5 \times 10^{-5}$  M). The air flow rate was adjusted to 0.3 and 0.5 dm<sup>3</sup>/min for conditioning and flotation stages, respectively. The concentrates and tails were dried and weighed, so as to calculate flotation recoveries for single minerals based on dry weights of the concentrate and feed. Flotation recoveries of artificially mixed minerals were calculated based on dry weights and grades of chalcopyrite and pyrite in the concentrate and feed. The grades of chalcopyrite and pyrite were obtained according to the chemical analysis of Fe and Cu based on stoichiometry formulae of CuFeS<sub>2</sub> and FeS<sub>2</sub> for chalcopyrite and pyrite, respectively. All tests were conducted in triplicates, with average recoveries reported. According to statistical analysis of the recovery data, the experimental error (relative standard deviation, RSD) for all tests was found to top at 3%.

#### Oxygen demand test

The oxygen demand tests were conducted in the micro-flotation cell (shown in Fig. 2) equipped with

an air purging system including a sintered glass-made sparger and an air valve connected to a compressed air cylinder. After purging air at 0.5 dm<sup>3</sup>/min for 2 min, the aeration was stopped for 2 min and maximum and minimum oxygen levels were measured as a function of time.

#### XPS Analysis

All samples were prepared under the desired conditions described in Section 3 and stored in liquid nitrogen to prevent further oxidation. XPS measurements were carried out on a physical electronic spectrometer (Specs Company, model EA10 Plus) using an Al K $\alpha$  X-ray source operating at 150 W. The pressure inside the analysis chamber was set to  $1.33 \times 10^{-7}$  Pa during the analysis. All spectra were calibrated using the carbon 1s spectral peak which has a known position of 284.6 eV.

## RESULTS AND DISCUSSION

### Flotation behaviour of single chalcopyrite, single pyrite, and their mixture

Before studying the effect of the ultrasonic treatment on the flotation separation of chalcopyrite from pyrite,

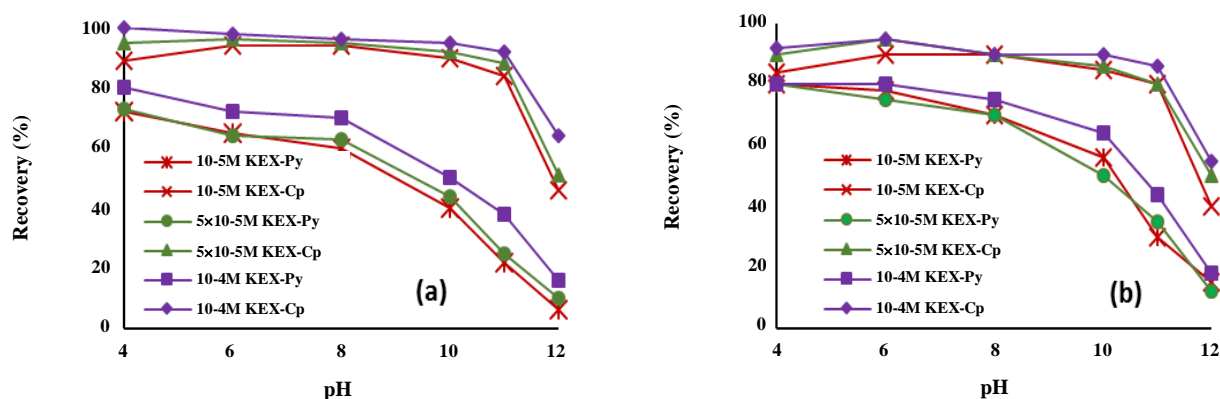


Fig. 3: Chalcopyrite (Cp) and pyrite (Py) recovery as a function of pH at different concentrations of KEX (a) single minerals (b) mixed minerals.

the floatability of single chalcopyrite, single pyrite, and their mixture was investigated to assess changes in recovery of each mineral in absence/presence of the other. Fig. 3 shows the flotation recoveries of the single minerals as well as their mixture as a function of pH at different concentrations ( $10^{-5}$ ,  $5 \times 10^{-5}$  and  $10^{-4}$  M) of KEX as the collector. Higher recovery of single chalcopyrite within a wide range of pH values, as observed in Fig. 3(a), may be attributed to the formation of hydrophobic metal xanthate or dixanthogen, while the decrease in recovery for pH values beyond 11 may be a result of the formation of metal hydroxide as a hydrophilic species. It was found that [15, 16] when the resting potential of mineral in solution is larger than the reversible potential of xanthate-dixanthogen couple, oxidation of xanthate to dixanthogen occurs and dixanthogen forms on mineral surfaces with the resting potential greater than 0.13 V. since the resting potential of pyrite is about 0.22 V, the flotation of pyrite under acidic conditions may be related to the formation of dixanthogen, as the main species responsible for the flotation of pyrite, while the decrease in recovery under alkaline conditions may be related to the formation of iron hydroxide species or lack of dixanthogen on the surface.

As shown in Fig. 3(b), flotation behaviour of the mixture of chalcopyrite and pyrite is, to some extent, different from the behaviour exhibited by the minerals in a single form. Indeed, chalcopyrite recovery decreases at all concentrations of the collector, with a partial increase in pyrite recovery. This may be attributed to the galvanic interactions occurring between these minerals in the

mixture. According to the literature [3, 17, 18], since pyrite is more cathodic than chalcopyrite, surfaces of pyrite particles may host an oxygen reduction reaction wherein metal ions from the surface of chalcopyrite are dissolved to form metal hydroxides on the chalcopyrite particles. Thus, the decrease in chalcopyrite recovery in mixed form may be a result of the formation of such hydrophilic species. On the other hand, the partial increase in pyrite recovery in the mixed form may be due to surface activation of pyrite particles by copper ions dissolved from the chalcopyrite surface [5, 19-21]. Note that, there may be a competition between chalcopyrite and pyrite to uptake the collector from solution. Since chalcopyrite may be floated by either of metal xanthate or dixanthogen but pyrite can only be floated by dixanthogen in the absence of a hydroxide/oxide layer, chalcopyrite, rather than pyrite, may uptake larger amounts of collector, while in the presence of hydroxide/oxide layers on chalcopyrite, pyrite may succeed to adsorb larger amounts of collector, making the pyrite more floatable.

Fig. 4 shows the Dissolved Oxygen (DO) profiles for the minerals, in single and mixed forms, as a function of time, when the oxygen is being either produced or consumed, with the air valve been turned on/off. The results of oxygen demand tests on the minerals in single and mixed forms (Fig. 4 (a)) show that the DO concentration in the suspension containing chalcopyrite only is greater than that of the suspension containing pyrite only, with the corresponding DO concentration to each of the minerals in the single form being greater than

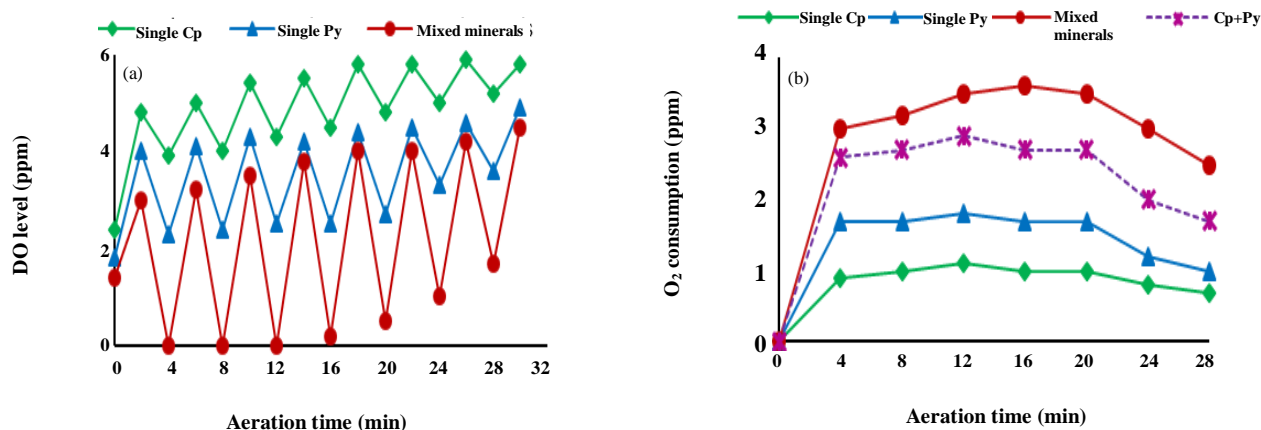


Fig. 4: Dissolved oxygen level variations (a) and oxygen consumption (b) as a function of time for chalcopyrite (Cp), pyrite (Py) and their mixture.

that of the mixed form at identical amounts of air supplied for 30 minutes. Indeed, the decrease in DO level when the air valve is closed demonstrates the mineral affinity towards oxygen consumption. Fig. 4(b) shows that the amount of oxygen consumed by the mixed minerals is more than the sum of oxygen consumed by both minerals when tested in single forms. The decrease in DO concentration for the suspensions of minerals in single and mixed forms when the air flow is stopped indicates higher oxygen demand of pyrite rather than chalcopyrite, because of further cathodic properties of pyrite. Further decrease in DO concentration for the suspension of the minerals in mixed form may be attributed to the galvanic interactions occurring between these minerals.

#### Effect of ultrasonic treatment on the separation of chalcopyrite from pyrite

According to the literature and the above-mentioned phenomena, interactions between chalcopyrite and pyrite make it difficult to separate chalcopyrite from pyrite in flotation industries. However, ultrasonic treatment of suspensions containing chalcopyrite and pyrite made it possible to float chalcopyrite and pyrite from suspensions containing the two minerals in either of single or mixed forms. Fig. 5 shows the results of the flotation separation of chalcopyrite from pyrite using ultrasonic treatment. As can be seen from Fig. 6, differences between the recoveries of chalcopyrite and pyrite after joint aeration and ultrasonic treatment are to some extent similar to

the differences between flotation recoveries of the single minerals. These results also indicate that ultrasonic treatment is more effective in alkaline conditions rather than acidic conditions. This may be related to the surface cleaning effect of ultrasonic waves that can remove metal hydroxide precipitates from the surface of chalcopyrite particles.

#### Surface characterization of the samples

XPS analysis was employed to identify surface species formed on the aerated mineral particles with or without ultrasonic treatment. Since significant changes in recoveries of the minerals were observed in alkaline conditions, pH of the samples was adjusted to 11 for XPS analysis. According to the literature [6, 22–25], the peaks around 705–708 eV in the Fe (2p) spectra are related to associated iron with S as in FeS, FeS<sub>2</sub> or CuFeS<sub>2</sub>, while the peaks around 710–713 eV and 724–726 eV represent iron hydroxide/oxide species. Also, the peaks at 932 and 952 eV in the Cu (2p) are related to copper sulphide mineral structures such as CuS species, while the peaks around 934, 935, and 942 eV represent copper oxide or hydroxide/oxide species. Fig. 7 shows the corresponding Fe (2p) and Cu (2p) XPS spectra for freshly mixed chalcopyrite and pyrite after 2 min of conditioning with nitrogen as an inert gas. It can be seen that almost all peaks are related to the mineral structures, except for a negligible trace of iron hydroxide/oxide species in Fe (2p) spectra around 724–725 eV.

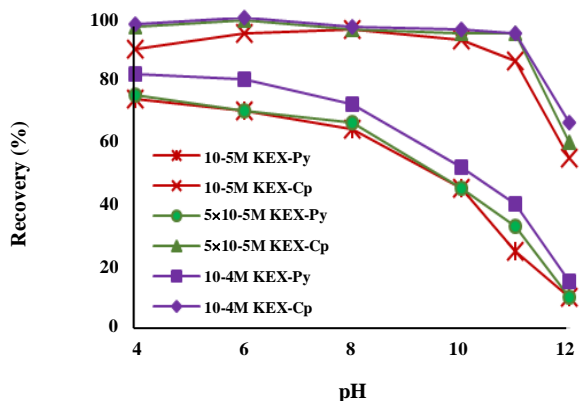


Fig. 5: Chalcopyrite (Cp) and pyrite (Py) recoveries as a function of pH at different concentrations of KEX after ultrasonic treatment.

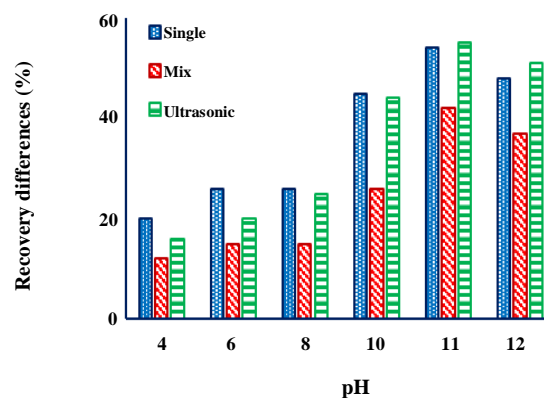


Fig. 6: Differences in chalcopyrite and pyrite recoveries as a function of pH at 10-4M KEX for suspensions containing the minerals in single and mixed forms and the ultrasonically treated suspension of the minerals.

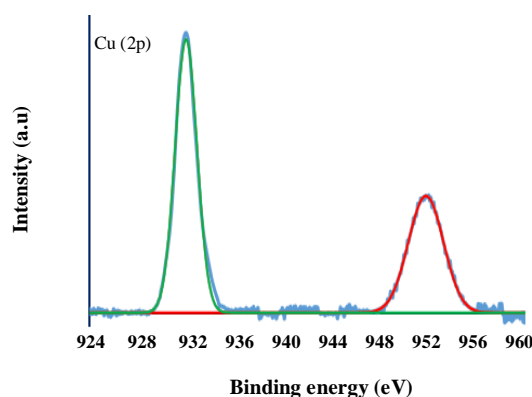
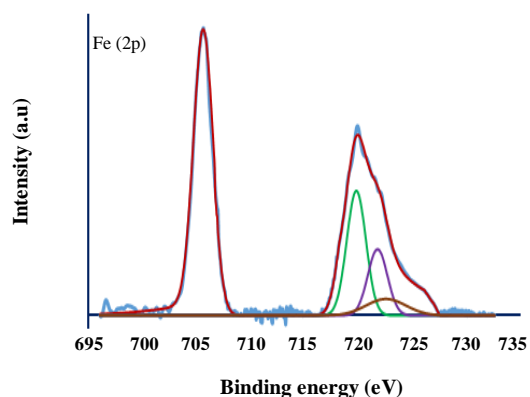


Fig. 7: XPS spectra of the mixture of chalcopyrite and pyrite conditioned with nitrogen gas at pH = 11.

Fig. 8 shows Fe (2p) and Cu (2p) XPS spectra for the mixture of chalcopyrite and pyrite once conditioned with aeration for 15 min. In this figure, some new peaks related to the metal (Fe and Cu) hydroxide/oxide species have appeared in either Fe (2p) or Cu (2p) spectra. According to the flotation results in Fig. 2 (b) in which the recovery of chalcopyrite is shown to decrease after aeration, almost all of the hydrophilic species may be formed on chalcopyrite particles. This may be a result of the galvanic interaction phenomenon in which the oxygen reduction and metal ions oxidation occur on the pyrite and chalcopyrite particles, respectively. This may result in the formation of hydrophilic metal hydroxide/oxide species on chalcopyrite particles. Thus, in the presence of such a hydrophilic layer on chalcopyrite, the further collector would be available to be adsorbed on the pyrite, increasing the pyrite recovery.

Fig. 9 shows the Fe (2p) and Cu (2p) XPS spectra for the mixture of chalcopyrite and pyrite after 15 min of conditioning and aeration along with ultrasonic treatment. As seen, most of the peaks related to hydroxide/oxide species have disappeared, with the corresponding peaks to the mineral structures appeared with slight changes. Considering the results of the flotation test shown in Fig. 4, it can be inferred that the mechanical vibration effect of ultrasound may result in the removal of colloidal hydroxide/oxide from the surface of the particles, making it possible to separate chalcopyrite from pyrite.

#### Effect of aeration on the flotation of the minerals

In order to investigate the effect of suspension aeration on the flotation of chalcopyrite and pyrite with/without ultrasonic treatment, some flotation tests

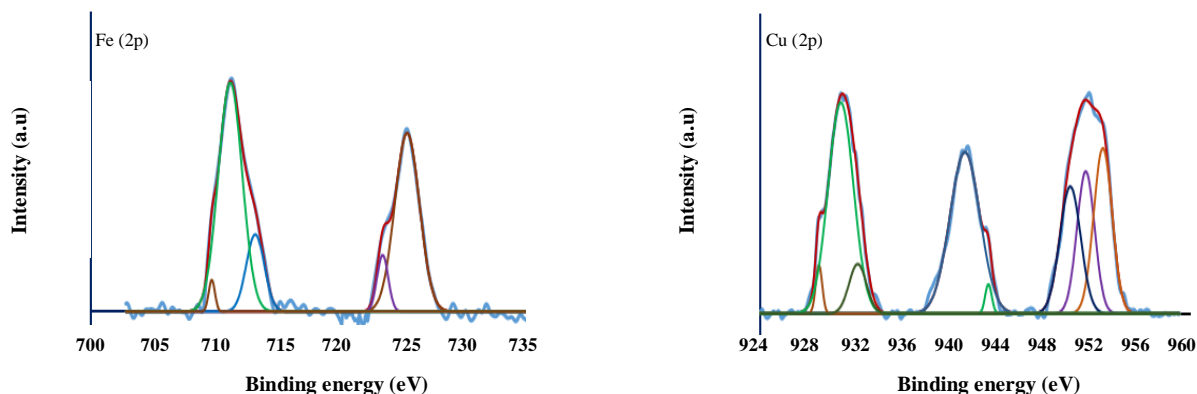


Fig. 8: XPS spectra of the mixture of chalcopyrite and pyrite after 15 min of conditioning and aeration at pH = 11.

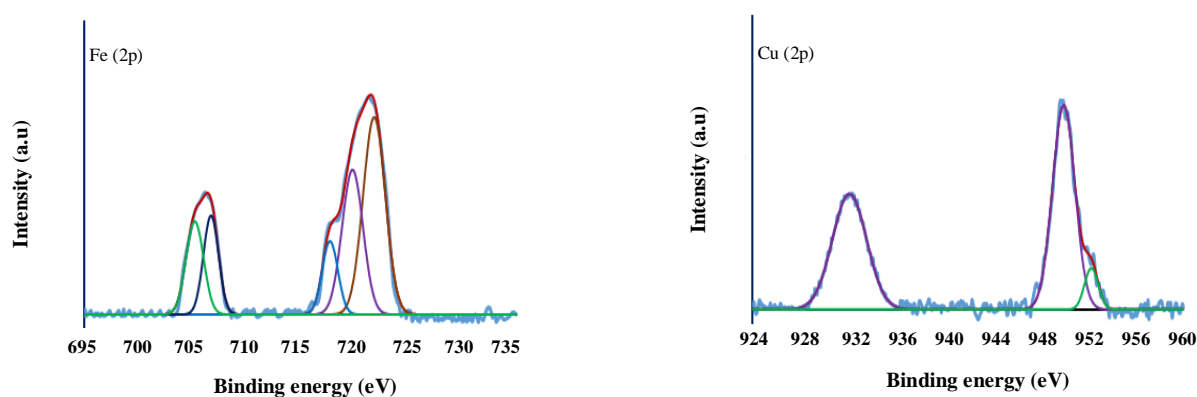
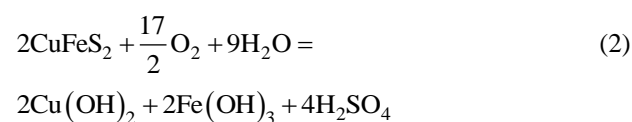


Fig. 9: XPS spectra of the mixture of chalcopyrite and pyrite after ultrasonic treatment at pH = 11.

were conducted with different aeration times. Fig. 10 shows the flotation recovery of chalcopyrite as a function of aeration time at different pH values (i.e. 6, 10 and 12) at  $10^{-4}$  M KEX for both of the cases with ultrasonic treatment and those without such treatments. As seen on the figure, chalcopyrite recovery decreases after 15min of aeration without ultrasonic treatment, especially at alkaline pH values, with no significant changes in chalcopyrite recovery with ultrasonic treatment. The decrease in chalcopyrite recovery without ultrasonic treatment at longer aeration times may be attributed to the formation of metal hydroxide species, as these hydrophilic species are not precipitated on chalcopyrite surface when subjected to the ultrasonic treatment.

Fig. 11 shows the flotation recovery of pyrite as a function of aeration time at different pH values (i.e. 6, 10 and 12) at  $10^{-4}$  M KEX for both of the cases with

ultrasonic treatment and those without such treatments. As can be seen, pyrite recovery follows almost similar trends with increasing the aeration time either with or without ultrasonic treatment. This indicates that the aeration does not impose any significant effect on pyrite flotation in the presence of chalcopyrite. In fact, most of the oxygen supplied by aeration is reduced on pyrite (Eq. (1)), forming hydroxide species on chalcopyrite (Eq. (2)); these hydrophilic species are then removed from chalcopyrite surface using ultrasonic treatment. These results are in agreement with the results found in XPS analyses.



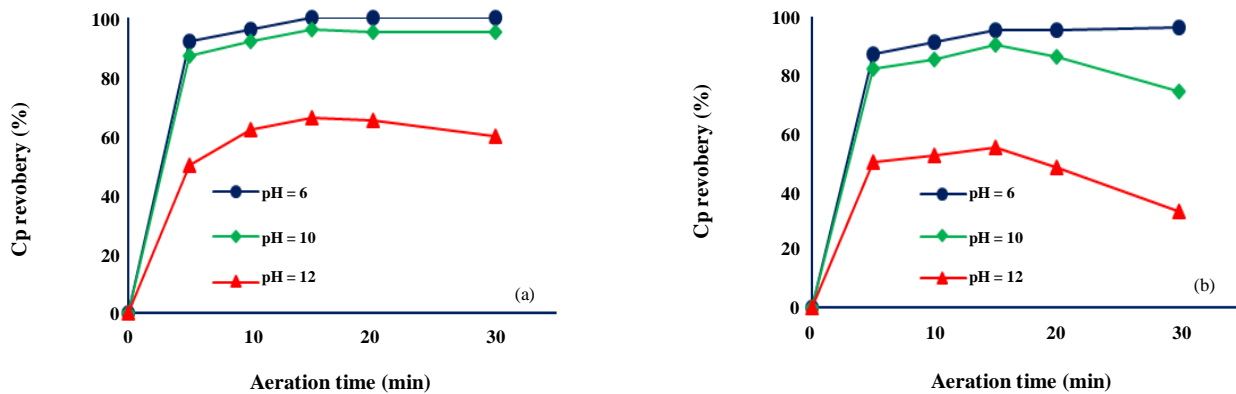


Fig. 10: Chalcopyrite (Cp) recovery as a function of aeration time (a) with or (b) without ultrasonic treatment at  $10^{-4}$  M KEX.

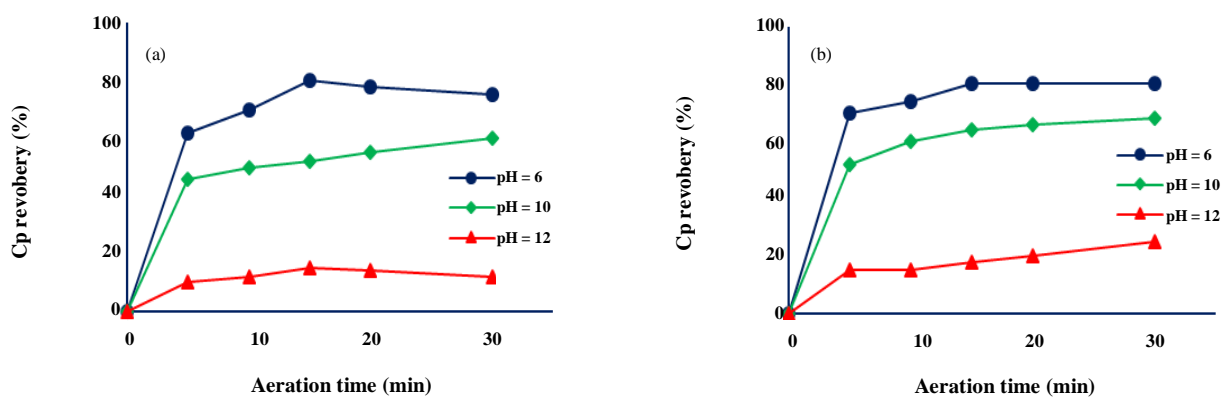


Fig. 11: Pyrite (Py) recovery as a function of aeration time (a) with or (b) without ultrasonic treatment at  $10^{-4}$  M KEX.

## CONCLUSIONS

Based on the experimental results, joint ultrasonic treatment and aeration enhanced the flotation separation of chalcopyrite from pyrite in artificially mixed mineral samples. Surface characterization by XPS analysis indicated that, compared to those conditioned without ultrasonic treatment, the mixed samples conditioned with ultrasonic treatment contained lower amounts of hydroxide/oxide species. This might be mainly due to the surface cleaning effect of ultrasound waves which may disperse the metal hydroxide/oxide precipitates from the surface of chalcopyrite particles, allowing the collector to be adsorbed on the particle surfaces. It seems that, in the mixed mineral samples, chalcopyrite and pyrite compete to adsorb the collector oxidation products. However, since the galvanic interactions between chalcopyrite and pyrite result in the formation of hydroxide/oxide species on chalcopyrite surface, most of the collector is adsorbed

on pyrite, making it more effectively floatable than chalcopyrite. But in the absence of hydroxide/oxide species, the collector is significantly adsorbed on chalcopyrite which can be floated by either metal xanthate or dixanthogen, as compared to pyrite which can be floated mainly by dixanthogen species only.

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