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Research Paper

Effect of Galvanic Interactions Between Grinding Media and Sulfide Minerals on Flotation Kinetics of Chalcopyrite

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Abstract: Although the flotation separation of sulfides and their electrochemical interactions have been examined in some investigations, study the direct relationship between the flotation kinetics of sulfides and their galvanic interactions during the process yet has not been addressed. To fill this gap, an extensive study was conducted to explore the effect of galvanic interactions for chalcopyrite and pyrite on their flotation kinetics when they are wetting ground by three different grinding media types (steel, chromium, and ceramic). Assessment of the flotation test results indicated that there was no galvanic interaction between the ceramic grinding balls and chalcopyrite. The galvanic interactions were mostly related to the grinding of chalcopyrite by steel balls. EDTA analysis showed that by increasing the amount of iron oxy/hydroxy and their precipitations on the surface of chalcopyrite, the recovery of chalcopyrite was decreased. Furthermore, increasing the pyrite content in the flotation of chalcopyrite increased the flotation rate constant and then decreased it. Flotation tests demonstrated that grinding chalcopyrite by ceramic media type, which has lower electrochemical activity in comparison with the other considered media, can be resulted in a higher flotation rate constant and ultimate recovery. Grinding the mixture of chalcopyrite and pyrite by steel or chromium media can show almost the same effect on the flotation rate constant and ultimate recovery. In general, the experimental results showed that by increasing the galvanic interaction, the flotation recovery and flotation kinetics were decreased.

Keywords: Galvanic interaction, Grinding balls, Chalcopyrite, Pyrite, Flotation recovery, EDTA extraction.

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1- INTRODUCTION

Galvanic interactions (GA) during grinding effectively can change the surface properties of sulfides and, as a result, change their floatabilities [1-4]. The conductivity characteristic of sulfide minerals facilitates electrons-transit into the aqueous solution between mineral sulfides and also between grinding media and minerals [5-7]. This electrons-transit increases the rate of GA, which can significantly diverse the flotation regime of sulfides. Thus, several investigations processed sulfide beneficiation, have been focused on this electrochemical term (Table 1). High variations in galvanic interactions can electrochemically generate high iron oxidation species, which increase corrosion of the medium [3,5,8].

Flotation kinetics, as a critical factor, can be used for evaluating and optimizing this separation process. Moreover, for designing flotation tanks, predicting metallurgical factors (recovery and grade), and scaling up, a determination of flotation kinetics trend plays a significant role. The recovery of mineral flotation is usually assumed as a first-order rate process, and it is represented as Equation 1.

$$R(t) = R_\infty\left(1 - e^{-kt}\right) \qquad (1)$$

Where:
R_t: the particle recovery at time t,
k: the flotation rate constant,
R_∞: is the flotation recovery at infinite time.

At different values of chemicals and physical operating conditions, different particles are responsible for different values of the equation parameters [14]. It was well documented that flotation kinetics depends on various chemical and physical operating conditions [15-18]. Kinetic models are useful to analyze flotation data and to evaluate the effect of operating conditions and also flotation chemicals [19]. However, indirect relationships between flotation kinetics and galvanic interaction have not been appropriately addressed. Therefore, to better understand possible interactions, this investigation is going to examine different types of galvanic interaction- (mineral-mineral interaction, mineral-grinding balls interaction, mineral-mineral-grinding balls interaction)- and for the first time (to our best knowledge) the effect of this galvanic interaction on the flotation kinetics.

2- MATERIALS AND METHODS

Ore samples (chalcopyrite and pyrite) were obtained from the Qaleh Zari mine (Birjand, Iran) and the Sungun copper mine (East Azerbaijan, Iran). The ore samples were ground to d_{80}=1300 µm by crushing in a jaw crusher, followed by dry grinding in a rod mill. They were divided by a riffle, and 100 g of samples were used for XRD and XRF analysis. The XRD results indicated that ore samples contained 27% chalcopyrite, and pyrite ores contained 58% pyrite. There were no other sulfide minerals in their compositions. The homogenized samples were sealed in polyethylene bags as ball mill feeds. The chemical compositions of ore samples were obtained by XRF (Table 2). The ball mill applied in all experiments was a standard bond ball mill obtained from BICO Company (USA), 305 mm×305 mm. Potassium Amyl Xanthate (PAX) (100 g/t) as a collector, MIBC (Methyl Isobutyl Carbonyl, 200 g/t) as a frother, and sulfuric acid (H2SO4, 0.5 mol/L) and sodium silicate (300 g/t) as depressants were considered for the flotation experiments. All inorganic reagents were AR grade and obtained from Merck Corporation (Germany). Sodium hydroxide was used for maintaining the pH in different experiments. High purity nitrogen, air, and deionized water were applied for flotation tests. EDTA (Ethylene diamine tetra acetic acid) was applied to determine copper and iron species. Three different types of balls, including steel, 13% chromium, and ceramic balls, were employed for grinding (Table 3). Three different diameters of grinding balls (3, 6, and 8 cm) were used in each grinding experiment with an equal ratio of every size. For minimizing the potential of contaminations, prior to each test, the mill was fed by pure silica, ran for around 30 min, and the whole system was purged by an air compressor to be cleaned and removed silica.

2-1- Galvanic interactions

In this study, the galvanic interactions between the grinding media and sulfide minerals were

Table 1. Various investigations on galvanic interactions and its effect on froth flotation

References	Procedures	Conclusions
Chen and Peng, 2015 [9]	A laboratory rod and stirred mill were used to regrind rougher flotation concentrates, and their effects on the separation of chalcopyrite from pyrite were explored.	Regrind with the rod mill produced more oxide species on chalcopyrite surface than that with the stirred mill. Oxidation of chalcopyrite produced a hydrophobic surface which promoted a collector-less flotation. Also, oxidation of pyrite hydrophilic species on the surface prohibited its activation by copper ions, resulting in low pyrite recovery. Thus, the highest level of separation achieved in the cleaner stage after regrinding with the rod mill
Azizkarimi et al., 2013 [6]	Influence of pH, immersion time, and applied potential on the chalcopyrite oxidation state were investigated	The resting potential of chalcopyrite increased by decreasing pH values. The rest potential of chalcopyrite electrodes increased sharply in the initial few seconds after the immersion and then continued at a slower rate due to the accumulation of oxidation products. The oxidation process occurred in three stages including, oxidation of top layer, deeper layer, and dissolution of passive layers
Allahkarami et al., 2014 [10]	The galvanic interactions between different grinding media (mild steel, cast iron, 10%chromium, 20%chromium, and ceramic media) and galena were investigated.	The floatability of galena is dependent on the galvanic current between grinding media and galena. High iron hydroxide species from the type of grinding media led to the lower flotation recovery of galena. Producing iron oxidation species for different types of grinding media had the following order: mild steel>cast iron> 10%chromium>20%chromium>ceramic
Yu et al., 2011 [11]	Electrochemical behavior of chalcopyrite in the presence of xanthate and the effect of potential on the surface film were studied by CV, SEM, and EIS	The hydrophobic film of X_2 had large thickness, the highest hydrophobicity degree, at the potential of 0 V. By increasing potential to 0.3 V, the surface film transformed to plenty of Cu(Ⅱ) and Fe(ⅡI) oxy-hydroxy species with high hydrophilicity
Peng and Grano, 2010 [3]	TOF-SIMS analysis for studying the effect of galvanic interaction (during grinding by different media) on flotation recovery of galena-pyrite and chalcopyrite-pyrite	Higher iron oxide species were seen on the chalcopyrite-pyrite surface than that on the galena-pyrite surface, since the more cathodic nature of chalcopyrite may draw more iron oxidation species from the anode, that is, grinding medium to the surface.
He et al., 2006 [12]	Separation of chalcopyrite from pyrite in different Eh values by depressing pyrite in the presence of zinc sulfate was investigated	The flotation recovery of chalcopyrite and pyrite is Eh dependent. At a fixed Eh of 275 mV when the efficiency was maximum, it was found that zinc sulphate depressed selectively pyrite at alkaline pH. Zinc sulfate was not able to depress pyrite at lower Eh values.
Huang and Grano, 2006 [13]	Using a Magotteaux mill to study galvanic interactions through grinding arsenopyrite by purging gas during different grinding media	Purging gas was not effective as grinding media. Purging oxygen gas caused the highest galvanic current in the medium-arsenopyrite couple. However, purging nitrogen gas resulted in the lowest regardless of the type of grinding medium.
Peng et al., 2003 [1]	Controlling pH throughout grinding by chromium and steel grinding balls, and investigating the effect of grinding media types on chalcopyrite flotation separation from pyrite.	Iron oxidation species and metal sulfide deficiency have an effect on chalcopyrite flotation. 30% chromium medium can increase the chalcopyrite recovery and improve selectivity.

examined in three interactions: mineral-mineral, mineral-grinding balls, and mineral-mineral-grinding balls.

2-1-1- Mineral-Mineral interaction

For investigating the effect of pyrite content on the pulp chemistry and chalcopyrite recovery (mineral-mineral interaction), pure chalcopyrite (-75 µm), and pyrite with (-150+90 µm) were provided from the ore samples (after initial grinding, pure mineral separated, and sieved) (Table 4). A 200 g mixed mineral system comprising of pure chalcopyrite and pyrite with different proportions of pyrite (0, 25, 50, and 75 wt.%) was placed into a 1.5 dm^3 (or 1500 cm^3) flotation cell (Denver). The sample was conditioned with the mentioned flotation condition. Flotation concentrates (different proportions of chalcopyrite and pyrite in 4 tests) were collected after a cumulative time of 0.5, 2, 5, and 8 min. The flotation froth was scraped every 10 s. Samples for EDTA extraction were obtained after each flotation test. An “88-mesh” screen was used to separate chalcopyrite and pyrite samples from flotation concentrate since they have different size fractions. Then, they were filtered, dried, and weighed for the cumulative recovery.

2-1-2- Mineral-Grinding balls interaction

The chalcopyrite ore sample (300g) was mixed with 1000 cm^3 of water, fed to the ball mill and ground by 5 kg of three different grinding medium types. Grinding continued for 30 min till 90 wt.% of the particles were less than 75 µm (P90: 75 µm). The pH during grinding was fixed at 11 by adding NaOH solution continuously. After grinding, samples for EDTA extractions were obtained from the mill chamber. Then, the slurry was pumped out of the mill and transferred to the 1.5 dm^3 (or 1500 cm^3) flotation cell (Denver). The samples were conditioned with the mentioned flotation procedure. Three flotation concentrates (derived by three different grinding media) were collected after a cumulative time of 0.5, 2, 5, and 8 min. The flotation froth was scraped every 10 s. The cumulative recovery was calculated for each weighed-dried fraction.

Table 2. Chemical compositions of chalcopyrite and pyrite ore samples

Composition	SiO_2	Al_2O_3	Fe_2O_3	Cu	S	P_2O_5	MnO	TiO_2	K_2O	Na_2O	CaO
Pyrite (wt. %)	7	3	38	0	50	0.09	0.02	0.07	0.53	0.18	0.52
Composition	SiO_2	Fe	MgO	Cu	SO_3	Pb	Co_3O_4	TiO_2	Ag	CaO	----
Chalcopyrite (wt. %)	58	12.20	0.70	15	13.20	0.01	0.30	0.56	0.03	0.13	----

Table 3. Chemical compositions of grinding balls (wt. %)

Composition	Cr	Fe_2O_3	C	Al_2O_3	Na_2O	SiO_2	Mn
Steel balls	1	94.6	2.5	-	-	1.1	0.8
Chromium balls	13	82.6	2.5	-	-	1.1	0.8
Ceramic balls	-	0.25	-	95	0.2	4.5	-

Table 4. Chemical compositions of chalcopyrite and pyrite for mineral-mineral interactions (chalcopyrite samples bore 95.6% chalcopyrite, and pyrite samples bore 98.6% pyrite)

Elements	Cu	Fe	SO_3	SiO_2	CaO	MgO	Pb	Co_3O_4	Ag
Chalcopyrite	33.10	29.20	34.60	1.90	0.13	0.51	0.01	0.30	0.03
Pyrite	0.15	45.90	52.60	0.00	0.15	0.57	0.01	0.04	0.02

2-1-3- Mineral-Mineral-Grinding balls interaction

A mixture of chalcopyrite and pyrite (150 g of each) from the ore samples with 1000 cm^3 water was placed into the mill and ground for 30 min using 5 kg of mild steel balls or chromium balls to achieve P90: -75 µm. The pH during grinding was maintained at 11 by pH titration through the continuous addition of NaOH solution. The samples for EDTA extractions were obtained from the chamber after grinding. The pulp was transferred to a 1.5 dm^3 (or 1500 cm^3) flotation cell (Denver) and processed with the above-mentioned flotation condition. Two flotation concentrates (2 different tests) were collected after a cumulative time of 0.5, 2, 5, and 8 min, and the flotation froth was scraped every 10 s.

2-2- Electrode's structure

Samples of pure chalcopyrite, pyrite, and grinding media were cut to a size of approximately 1 cm^2. A copper wire was connected to the prepared minerals or grinding media specimen through electrically conductive silver epoxy. The electrodes were made up by setting them in a non-conductive resin epoxy and resin hardener. Before each experiment, the electrode surface was polished by abrasive 600-grade sandpaper and then washed with deionized water and transferred immediately to the electrochemical cell for the rest potential measurement. All resting potential measurements were performed with a reference electrode of Ag/AgCl. For determining the galvanic properties of minerals and grinding balls through their electrodes, the Eh changes versus pH were recorded [3]. The resting potential of electrodes was measured to compare the electrochemical reactions of mineral and grinding balls. As a result, the cathodic and anodic reactions in the galvanic couple were determined. The resting potential of all electrodes was explored in the electrochemical cell consisting of water with pH 3, 7, 9, or 12. The resting potential of chalcopyrite or pyrite electrodes was also recorded in the pulp pH of 7 and the presence of 150 g/t collector.

2-3- Eh and pH variation

Eh and pH variation as indicative factors of interactions between minerals, or between minerals and grinding media were measured. Study the trend of these variations through galvanic interactions that may lead to a deep understanding of the process, the results of galvanic coupling, and, finally, better control of galvanic interactions. To this end, the redox potential (Eh) and pH of pulp were measured during 20 min of grinding 300 g chalcopyrite by the three mentioned types of grinding balls with the initial pH at 11, which was adjusted by adding NaOH solution.

2-4- EDTA extraction technique

EDTA extraction technique was employed to determine the magnitude of oxidized iron species in slurries [20] and to measure system corrosion [21]. A 3 wt. % solution of analytical (AR) grade ethylene diamine-tetra acetic disodium salt was prepared, and NaOH was used to adjust the pH at 7.5-8. The EDTA solution had been continuously purged by nitrogen for around 2 h, before running the experiments (due to eliminating oxygen in the solution as well as atmosphere). The obtained sample from the slurry (about 10 ml) was purged by nitrogen and filtered through the "0.45 µm Whatman filter". The weighed and dried filter cake was added to the prepared EDTA solution, whereas nitrogen was continuously purged throughout in a magnetically stirred vessel. Accordingly, the filter cake was leached for 10 min. Then, the EDTA extracted slurry filtered through a "0.45 µm Whatman filter". The two filtrates' solutions were weighed and analyzed for determining the iron and copper contents by AAS. The filter cake was also dried and weighed to calculate the mass of oxidized metal species.

2-5- Kinetic studies

The effect of galvanic interactions associated with the grinding media and the flotation cell on the kinetics of flotation separation was investigated. In each flotation test, the froth concentrates were collected after a cumulative time of 0.5, 2, 5, and 8 min. Three typical first-order kinetic models (Table 5) were fitted to the experimental recovery-time curves. The first phase of the kinetic study is to determine the best-fitted flotation model based on the experimental recovery-time curve. The second phase of the kinetic assessment is to explore the influence of galvanic interactions on the kinetic parameters, including flotation rate constant and ultimate recovery, and the modified rate constant, which are derived from the best-fitted model. Table 5. Applying kinetic models and implanted in the statistical software (Mathematical software, Wolframe 2000). R is the recovery of components at a specific time, R∞ is the ultimate recovery and represents the efficiency of flotation, and k refers to the flotation rate constant [22]. The rate constant is composed of different parameters, including the dynamics of flotation cell and mineral properties.

Table 5. Kinetic models (R is the recovery of components at a specific time, R∞ represents the ultimate recovery, and k refers to the flotation rate constant [22])

Model	Equation
Classic	$R = R_{\infty} * (1 - e^{-kt})$
Klimpel	$R = R_{\infty} * (1 - e^{-kt}) * (1 - \left(\frac{1}{kt}\right))$
Fully mixed reactor	$R = R_{\infty} * (1 - \frac{1}{1 + \left(\frac{t}{k}\right)})$

3- RESULTS AND DISCUSSION

3-1- pH vs. resting potential

Assessments of the open circuit potential can be used to determine the role of minerals and mediums in galvanic reactions. In terms of thermodynamics, the higher resting potential represents the lower electrochemical reactivity (the noble material). The electrochemical activity demonstrates which mineral acts as an anode or a cathode during the galvanic interactions. A mineral with higher resting potential (noble mineral) acts as a cathode and the other with lower resting potential (active mineral) as an anode electrode [3]. Assessing the resting potential of the performed experiments (Figure 1) shows that pyrite is nobler than other materials. The resting potential has the following order: pyrite> chalcopyrite> chromium> mild steel electrodes (Figure 1). This order can be translated as pyrite having the least electrochemical activity. Thus, through the preformed experiments, pyrite constantly played the role of a cathode in the galvanic interactions. This assessment also indicates that chalcopyrite is being more active than pyrite (which has a lower resting potential) and acts as an anode in the galvanic coupling with pyrite. Therefore, the galvanic interaction between chalcopyrite and pyrite is spontaneous.

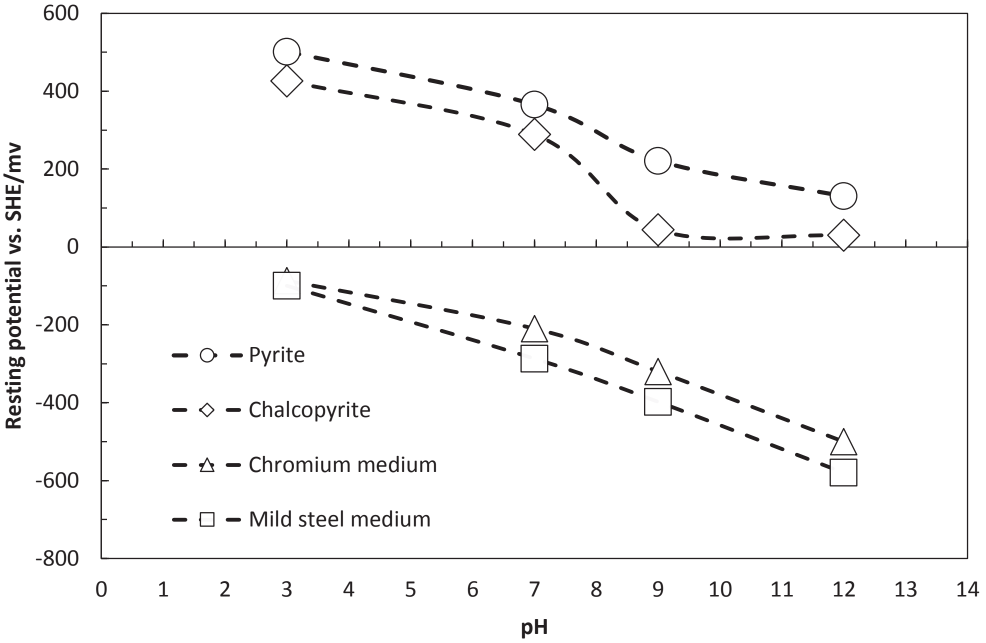


Figure 1. The diagram of open circuit potential for different minerals and grinding balls

The chalcopyrite, which is more active, serves as an anode where oxidation takes place as follows [23-26]:

$$Fe^0 + 2OH^- = Fe(OH)_2 + 2e^- \quad (2)$$

$$Fe(OH)_2 + OH^- = Fe(OH)_3 + e^- \quad (3)$$

While pyrite serves as a cathode, on which reduction reactions take place:

$$O_2 + 2H_2O + 4e^- = 4OH^- \quad (4)$$

These oxidation-reduction reactions lead to the dissolution of the chalcopyrite surface.

In a ball mill, minerals and grinding balls are constantly in contact, which affects the galvanic current between mineral samples and the medium. Accordingly, ores and grinding balls with the highest potential difference cause the most corrosion for the grinding balls [27]. As the experimental results show (Figure 1), the most resting potential difference is related to the pyrite and steel grinding balls. Therefore, the strongest galvanic interaction occurs when they are coupled in a galvanic medium. The resting potential of the chalcopyrite-pyrite mixture is higher than the potential of chalcopyrite and lower than the potential of pyrite [23,28].

The result of measuring electrode potential for chalcopyrite and pyrite at pH 7 and in the presence of xanthate collector also proved that pyrite is constantly cathode with higher potential recorded. The resting potential of pyrite and chalcopyrite was recorded at 0.295 and 0.19 mv, respectively. Thus, even in the presence of the collector, the resting potential of pyrite is constantly more than chalcopyrite (as mentioned, pyrite plays the role of the cathode in the galvanic interaction with chalcopyrite). The product of this interaction is dixanthogen based on the following equations:

$$2x^- \rightarrow x_2 + 2e^- \quad (5)$$

The formation of dixanthogen is:

$$MS + 2x^- \rightarrow Mx_2 + S + 2e^- \quad (6)$$

$$H_2O + 2e^- \rightarrow 2OH^- \quad (7)$$

3-2- Grinding types

pH assessments during grinding of chalcopyrite by using different balls show (Figure 2) that pH constantly decreases through the process for all types of considered grinding balls. This decrease can be due to the fact that based on Equations 1 and 2, OH- ions form into iron hydroxides. Reduction in the pH values makes corrosion (which is evident in the acidic pulp), and the rate of corrosion amount increases. Therefore, factors, which decrease the pH of pulp, can increase the rate of corrosion in the ball mills. The corrosion during grinding can affect the subsequent flotation recovery. This corrosion is why controlling pH during grinding sulfide ores is a necessary process [27,28]. The pH assessments also (Figure 2) indicate that using steel grinding balls can lead to the lowest measured pH values between the preformed experiments. While employing the ceramic grinding balls does not show a significant variation in the pH values, which can be as a result of no galvanic interaction between the ceramic grinding balls and chalcopyrite. Also, the pH changes in grinding chalcopyrite by mild steel media and chromium media have shown almost the same trend in 14 minutes, but the galvanic interaction of which contributes to their difference in pH changes in the minute of 20.

Nevertheless, by decreasing pH values, it is expected that the production of H+ increases, which, in turn, tends to increase the oxidizing environment or increase in Eh values. However, concerning the changes in pH from 11 to 8 (Figure 2), which still are in the range of alkaline, the production of oxidizing environment was limited. Similarly, this trend has been introduced in other investigations [29]. Eh variations during grinding chalcopyrite by different media types (Figure 3) show that grinding chalcopyrite by ceramic balls does not affect the Eh trend (Eh of pulp before and after grinding is approximately the same). This is because no galvanic interaction occurs between ceramic balls and chalcopyrite during grinding, while steel balls change the pulp Eh to negative quantities through grinding due to the presence of air, and, correspondingly, limiting oxidizing environment. Chromium balls also decrease the initial pulp Eh from 73 mv to around 0 mv (Figure 3). The reduction in the pulp Eh is due to oxygen consumption through the cathodic process (Equation 3). These galvanic interactions continue until the entire oxygen of the pulp is hydrolyzed. Afterward, the interaction will be stopped. During grinding sulfide ores, the oxidation-reduction (mostly made by grinding media) changes the surface properties of particles and affects the following process recovery (i.e., flotation). This

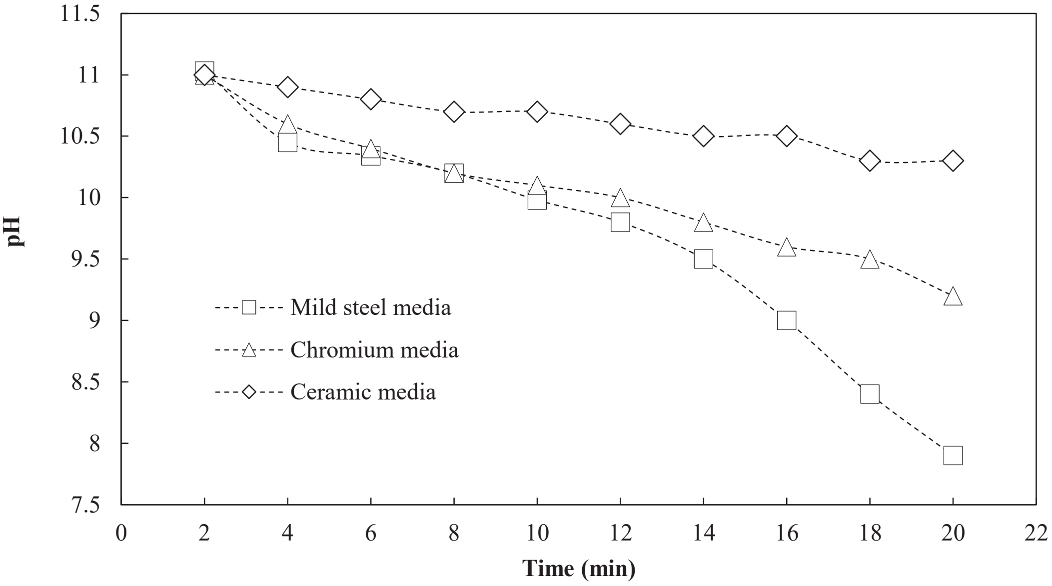


Figure 2. pH variations during grinding chalcopyrite by different grinding balls

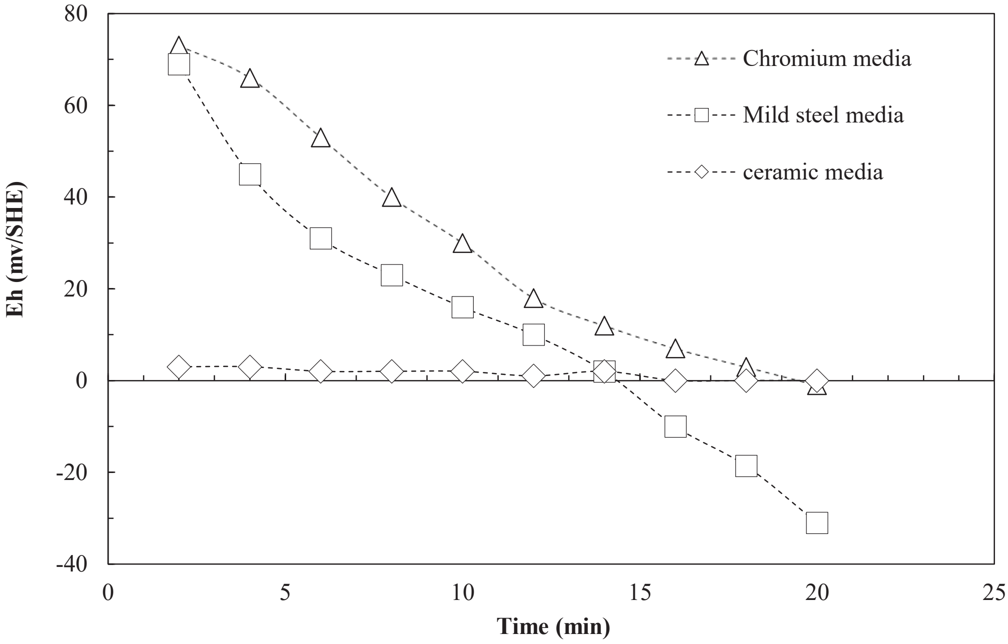


Figure 3. Eh variations during grinding chalcopyrite by different grinding balls

phenomenon will affect the collector adsorption (typically xanthate) on the sulfide surface, which is an electrochemical reaction, including using oxygen in the cathodic reactions (Equations 4-6) [30]. By completely utilizing oxygen in the grinding process, a low volume of oxygen remains to form metal xanthate or dixanthogen. Thus, the adsorption of xanthates on the mineral surface does not efficiently occur [30,31], and the flotation recovery will be significantly affected. In a word, floating sulfide ores heavily depend on galvanic interactions that occurred through the grinding step.

3-3- Galvanic interactions

3-3-1- Mineral-Mineral interaction

It was well documented that the flotation separation of chalcopyrite from pyrite depends on oxide species formed on the surface of these minerals. Therefore, in order to reach an optimum separation efficiency, generating iron and copper oxides should be controlled by providing an effective flotation environment [32]. Determination of the pure chalcopyrite cumulative recoveries (Figure 4) (in the presence of different pure pyrite percentages during the performed experiments) shows that by increasing the pyrite content in the mixture from 0 to 75%, the recovery of chalcopyrite decreases from 88 to 22%. In other words, the recovery of chalcopyrite lowered as the pyrite content in the mixture increased. This phenomenon can be a result of higher pyrite amounts in the mixture, which may cause higher galvanic interactions (Figure 4) and reduce the chalcopyrite recovery.

EDTA extraction for assessing mineral-mineral interactions (Figure 5) shows that the concentration of iron species increases and the concentration of copper species decreases when the percentage of pyrite in the mixture has increased. The most extracted copper species is related to the floating single chalcopyrite since there were not any oxidant agents causing the depression of copper species in this experiment (Figure 5). The lowest detected value of iron species is observed in the flotation of single chalcopyrite. The iron extracted in this flotation test is mainly derived from the dissolution of chalcopyrite particles. When pyrite is in the mixture, the iron species extract from chalcopyrite dissolution. Moreover, oxidation-reduction is made by chalcopyrite and pyrite galvanic interaction. To such an extent, the most extracted iron species are detected for the mixture of 75% pyrite and 25% chalcopyrite. This enhancement in the concentration of iron species can be explained by the decrease of chalcopyrite recovery (Figure 5). As a result of

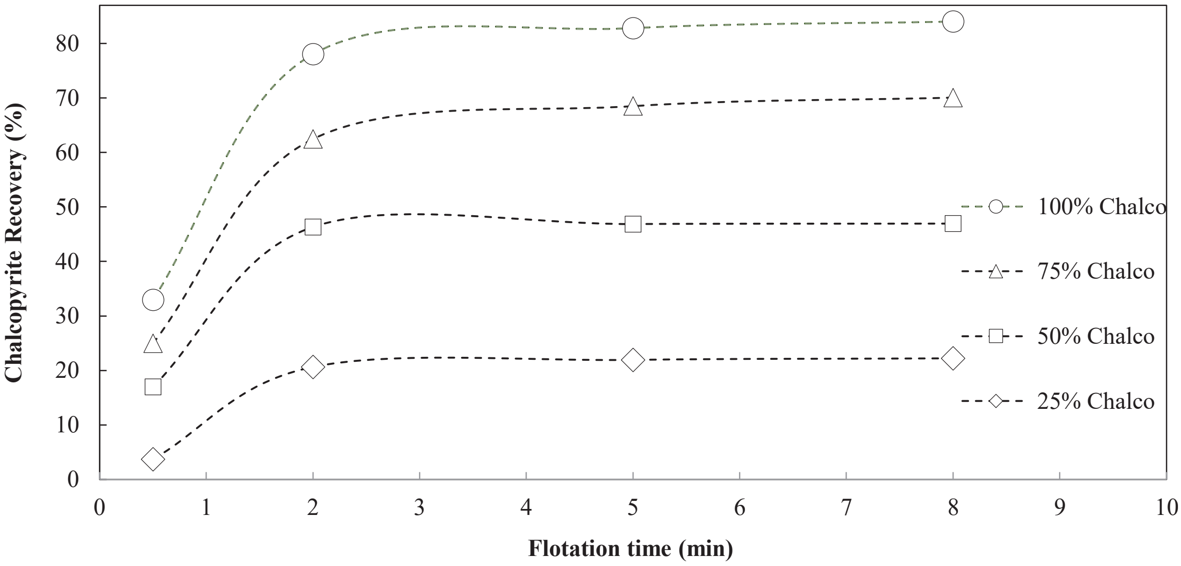


Figure 4. Effect of chalcopyrite-pyrite contact on chalcopyrite flotation

galvanic interactions, hydroxide species formed through hydrolyzing iron species; since then, they precipitated on the chalcopyrite surface and turned it into a hydrophilic surface [33]

3-3-2- Mineral-grinding balls interaction

Exploring the effects of different grinding balls on chalcopyrite recovery (Figure 6) when the grounded ore was considered (27% chalcopyrite and 59% pyrite) demonstrates that the maximum chalcopyrite recovery is occurred for samples ground by ceramic balls. The minimum chalcopyrite recovery is related to the ground samples by steel balls.

Hydroxide iron species produced during grinding chalcopyrite in the galvanic environment play an important role in lowering the recovery; since they are hydrophilic ions, precipitate on the

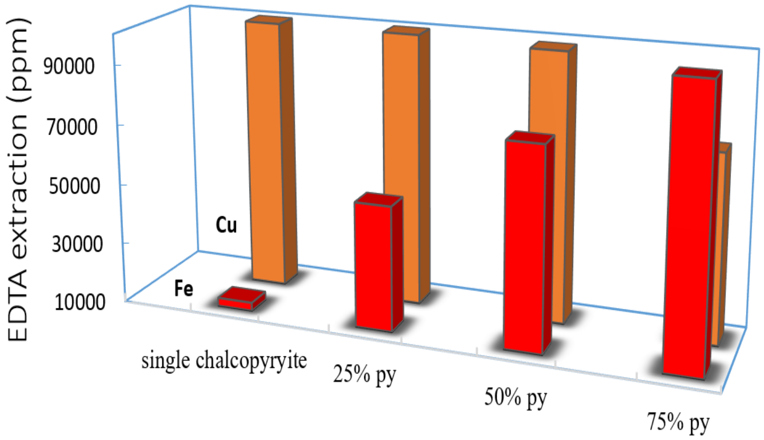


Figure 5. EDTA extractable iron and copper under different grinding conditions (single chalcopyrite is 100% chalcopyrite, 25% pyrite is 75% chalcopyrite + 25% pyrite, 50% pyrite is 50% chalcopyrite+ 50% pyrite, 75% pyrite is 25% chalcopyrite + 75% pyrite)

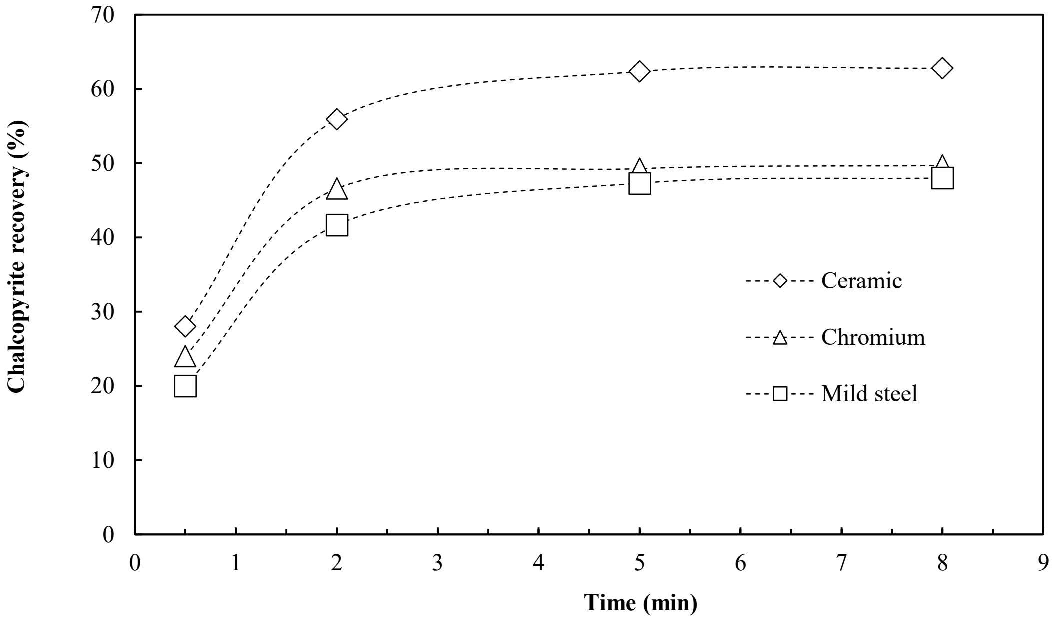


Figure 6. Effect of different grinding balls on chalcopyrite flotation as a function of time

mineral surfaces, and hinder them from floating [33]. While, according to the pourbaix diagram, hydroxide species of copper ions in sulfide solution include Cu_2S, CuS, Cu(OH), Cu_2O, CuO, $Cu(SO_4)$, $Cu_4So_4(OH)_6$. These oxy-hydroxy metal species precipitate on the mineral surface and prevent it from subsequent corrosion. Half reactions in which metal is oxidized in alkaline media are as follows [30,34]:

$$CuO + 2H^+ + 2e^- \rightarrow Cu + H_2O \quad (8)$$

$$Cu_2O_3 + 2H^+ + 2e^- \rightarrow 2CuO + H_2O \quad (9)$$

$$2CuO_2 + 2H^+ + 2e^- \rightarrow Cu_2O_3 + H_2O \quad (10)$$

$$Cu_2O_3 + 6H^+ + 2e^- \rightarrow 2Cu^{2+} + 3H_2O \quad (11)$$

$$CuO_2 + 4H^+ + 2e^- \rightarrow Cu^{2+} + 2H_2O \quad (12)$$

EDTA extraction (Figure 7) shows the effect of grinding conditions on the magnitude of extracted iron and copper in the slurry. As outcomes illustrate, the grinding media have different influences on the amount of EDTA extracted species (Figure 7). Steel grinding balls produce the highest and the lowest amount of EDTA extracted iron and copper in the slurry, respectively. While ceramic grinding balls produce the highest and the lowest amount of EDTA extracted copper and iron by far. In other words, the fewer electrochemical reactions during grinding can lead to less amount of EDTA extractable iron. The high amount of EDTA extractable copper in the mill discharge is in an appropriate agreement with the chalcopyrite recovery.

3-3-3- Mineral-Mineral-Grinding balls interaction

The effect of grinding samples by two different balls on chalcopyrite flotation in the mixture of chalcopyrite and pyrite (Figure 8) demonstrates that both chromium and steel grinding balls produce almost the same amount of chalcopyrite recovery. In terms of electrochemistry, grinding balls are more active than minerals, thus acting as an anode on which oxidation occurs [1,3,8]. On the other hand, pyrite is the noblest mineral and plays the role of a cathode, where the reduction takes place. Chalcopyrite, with the intermediate magnitude of rest potential between pyrite and grinding balls, may play both roles of oxidation and reduction simultaneously.

Assessing the EDTA extracted amounts of iron and copper in the chalcopyrite-pyrite mixture experiments (Figure 9) shows through grinding by steel grinding balls, the extracted amounts for both metals are much higher than grinding by chromium balls. Different investigations with various conditions such as different solid percent, the size of the mill, the aeration, the type of mineral, the effect of minerals in the multi-mineral system, etc. can directly affect the number of ion species

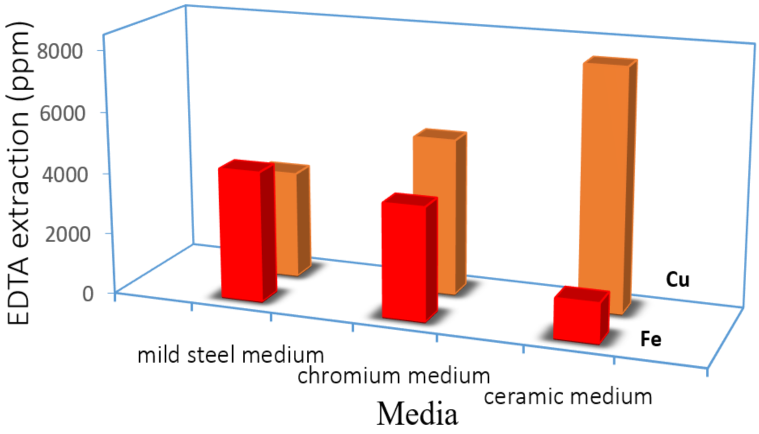


Figure 7. EDTA extractable iron and copper under different grinding medias

extracted by EDTA. The presence of pyrite in the mixture can be the main reason why pyrite is the noblest mineral and has the most difference in the resting potential with the steel media. As a result, galvanic current and electrochemical activity in the slurry increases, and makes more dissolution of mineral surfaces (the amount of extracted ions enhances). However, copper ions may not float since they are depressed by iron hydroxides due to the high magnitude of the presented iron species. These phenomena can explain the lower flotation recovery of chalcopyrite through grinding by steel balls compared to chromium balls. Besides, galvanic interactions between noble (pyrite) and active (chalcopyrite) minerals can principally influence the floatability of the active mineral more than the noble one. Therefore, applying grinding balls with more chromium content (for example, more than 13%) can produce oxide species to the extent that depress pyrite minerals. However, those oxide species are not enough to depress chalcopyrite minerals. Thus, they enhance chalcopyrite recovery since there could be sufficient oxide species in the pulp, which can hinder the activation of pyrite minerals by copper ions [23,24].

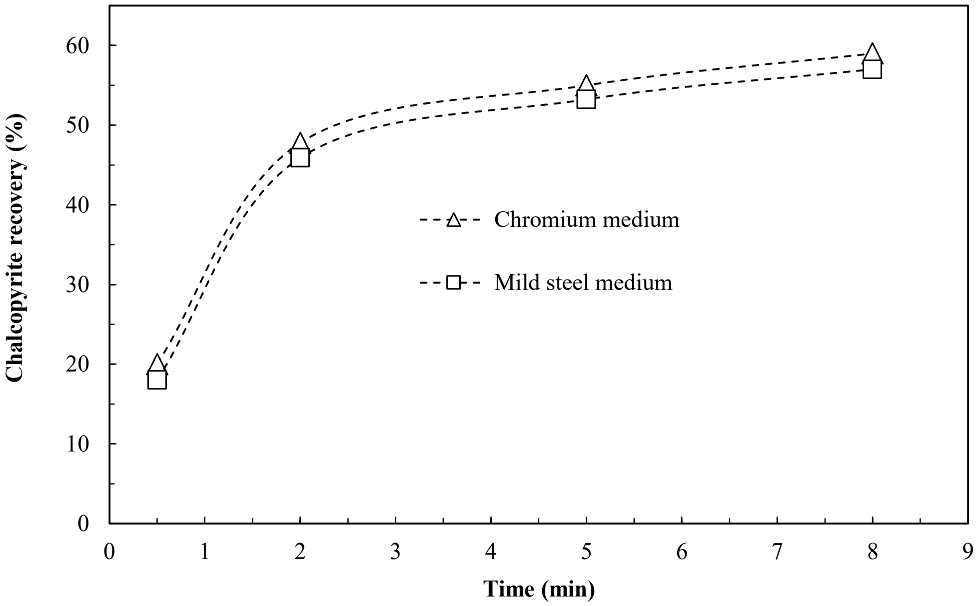


Figure 8. Effect of grinding media on chalcopyrite flotation in the mixture of chalcopyrite-pyrite

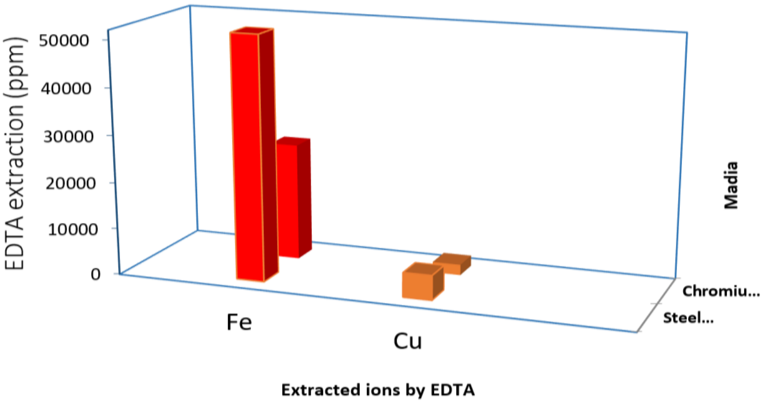


Figure 9. EDTA extractable iron and copper under different grinding conditions in grinding chalcopyrite-pyrite mixture

3-4- The flotation kinetic

Different kinetic models are fitted to the data to find the most representative model and assess the relationship between the flotation rate constant (k) and various galvanic interactions. According to the results (Tables 6-8, and Figure 10), the best-fitted model to the experimental data is the classic model. Since the classical model fitted the experimental data adequately, this model was utilized to investigate the effect of different galvanic conditions on flotation kinetics.

The kinetic parameters ,R ,∞k ,and K_m based on the classic model, are presented in Table 9. The relationships between flotation responses and galvanic interactions for different conditions are displayed in Figures 11-13.

Table 6. Experimental data versus recoveries estimated by kinetic models (chalcopyrite-pyrite galvanic interactions)

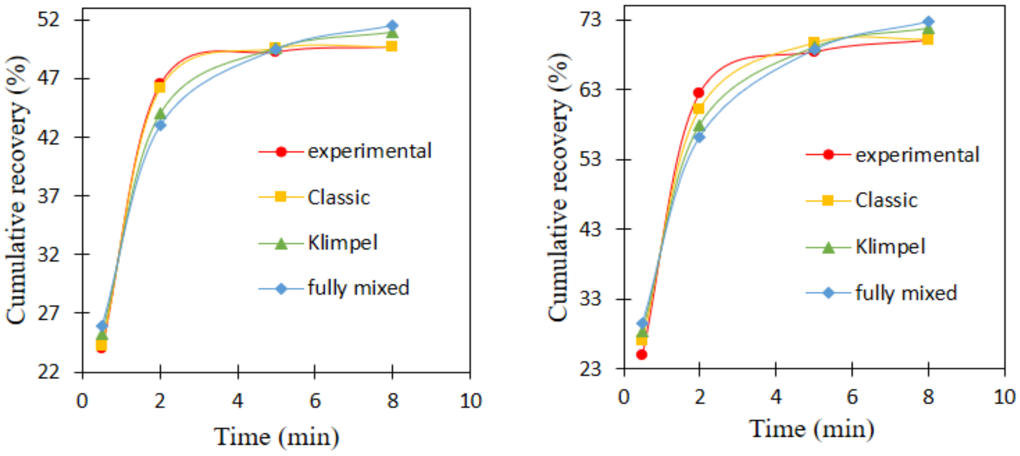
Condition	Time	Experimental	Classical	Klimpel	Fully mixed reactor
100%Chalcopyrite	0.5	33	35.4	36.9	38.4
	2	78.1	74.9	71.8	69.8
	5	82.8	84.2	83.7	83.4
	8	84.1	84.5	86.8	87.7
75% Chalcopyrite-25% Pyrite	0.5	25.0	27.0	28.3	29.5
	2	62.5	60.2	57.9	56.3
	5	68.5	69.7	69.1	68.8
	8	70.1	70.2	71.9	72.8
50% Chalcopyrite-50% Pyrite	0.5	17.0	20.0	20.6	21.5
	2	46.3	42.5	40.6	39.5
	5	46.9	47.9	47.6	47.4
	8	47.0	48.1	49.4	49.9
25% Chalcopyrite-75% Pyrite	0.5	3.7	7.2	7.3	7.6
	2	20.6	17.8	17.2	16.6
	5	21.9	22.4	22.1	21.9
	8	22.2	22.8	23.4	23.8

Table 7. Experimental data versus recoveries estimated by kinetic models (grinding chalcopyrite by several grinding media)

Condition	Time	Experimental	Classical	Klimpel	Fully mixed reactor
Mild steel medium	0.5	20.0	19.7	20.8	21.6
	2	41.7	42.0	40.4	39.3
	5	47.3	47.4	47.1	47.0
	8	48.0	47.7	48.8	49.4
Chromium medium	0.5	24.0	24.2	25.2	25.9
	2	46.6	46.2	44.1	43.0
	5	49.3	49.6	49.6	49.5
	8	49.7	49.7	51.0	51.5
Ceramic medium	0.5	28.0	27.6	29.0	30.1
	2	55.9	56.5	54.1	52.7
	5	62.4	62.3	62.1	62.0
	8	62.8	62.5	64.1	64.7

Table 8. Experimental data versus recoveries estimated by kinetic models (grinding chalcopyrite-pyrite by several grinding media)

Condition	Time	Experimental	Classical	Klimpel	Fully mixed reactor
Mild steel medium	0.5	18.0	18.7	19.7	20.7
	2	45.9	45.0	43.7	42.5
	5	53.2	54.9	54.3	53.9
	8	57.0	55.8	57.1	57.8
Chromium medium	0.5	20.0	20.2	21.3	22.3
	2	47.8	47.3	46.0	44.7
	5	55.0	56.8	56.2	55.9
	8	59.0	57.5	58.8	59.6



(A) (B)

Figure 10. Comparison of the first-order kinetic models fitted to the experimental flotation recovery data A: Grinding chalcopyrite by chromium grinding balls B: Chalcopyrite-pyrite galvanic interactions, including 75% chalcopyrite and 25% pyrite

Table 9. Estimated kinetic parameters by the classic model

Condition		R_∞ (ultimate recovery), %	k (flotation rate constant), min^{-1}
Chalcopyrite-pyrite galvanic interactions	100% ch	84.6	1.08
	75% ch- 25% py	70.2	0.97
	50% ch- 50% py	48.1	1.17
	25% ch- 75% py	22.9	0.75
Grinding chalcopyrite by distinct media	Mild steel medium	47.7	1.07
	Chromium medium	49.7	1.34
	Ceramic medium	62.5	1.17
Grinding chalcopyrite-pyrite by various media	Mild steel medium	55.9	0.82
	Chromium medium	57.6	0.86

Assessment of modeling outcomes (Figure 11A) shows an increase in the k by increasing the chalcopyrite amount in the mixture from 25 to 50%. While, by increasing chalcopyrite in the mixture of chalcopyrite-pyrite from 50 to 75%, k is decreased. This phenomenon could potentially be a result of pyrite activation by copper cations. The presence of pyrite (pyrite having the highest rest potential of the common sulfide) may enhance the oxidation of other presented less noble metal sulfide, and lower the selectivity. Results (Figure 11B) also indicate that by increasing the percentage of chalcopyrite in the mixture (the same solid content), the ultimate recovery is increased.

Exploring the flotation rate constant when different mineral-grinding types are used (Figure 12A) indicated that grinding chalcopyrite by chromium balls has the highest k among the experiments. While grinding by mild steel medium shows the lowest k. By applying ceramic balls instead of chromium grinding balls, k has decreased. Producing iron oxidation species through grinding chalcopyrite by ceramic balls in iron mill space can answer this decrease. The maximum and the minimum percentage of ultimate recovery obtain in grinding chalcopyrite by ceramic and mild steel grinding balls, respectively (Figure 12B). Grinding by mild steel and chromium balls produces a negligible difference in both k and ultimate recovery (Figure 13). A similar trend of

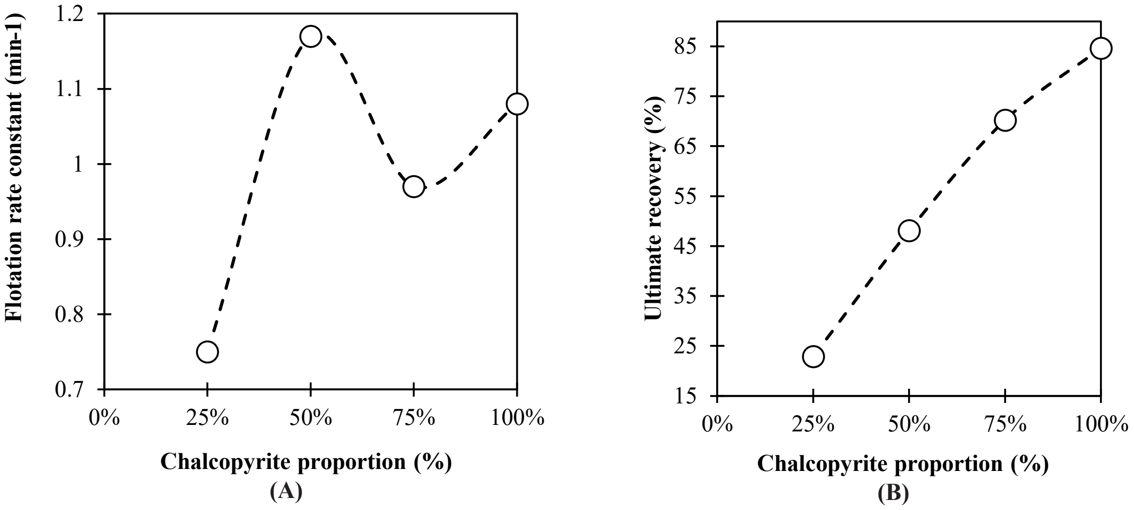


Figure 11. Effect of chalcopyrite-pyrite galvanic interactions on A: Flotation rate constant and B: Ultimate recovery

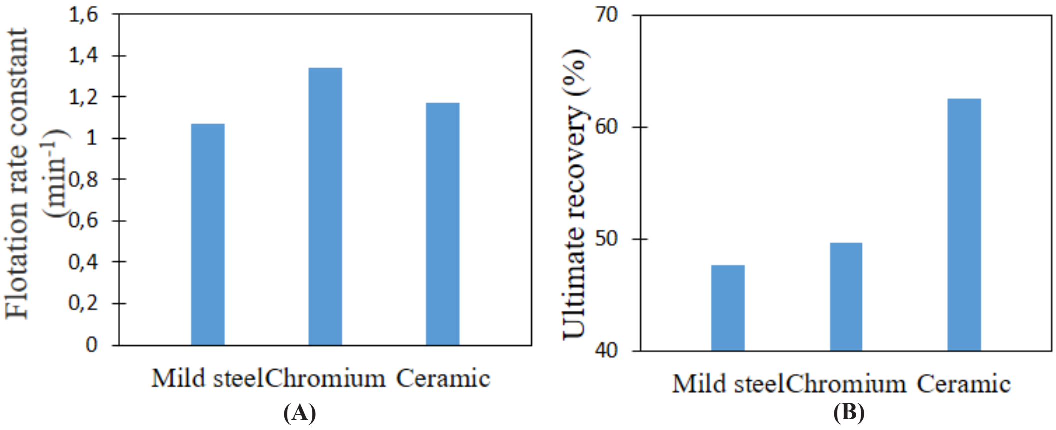
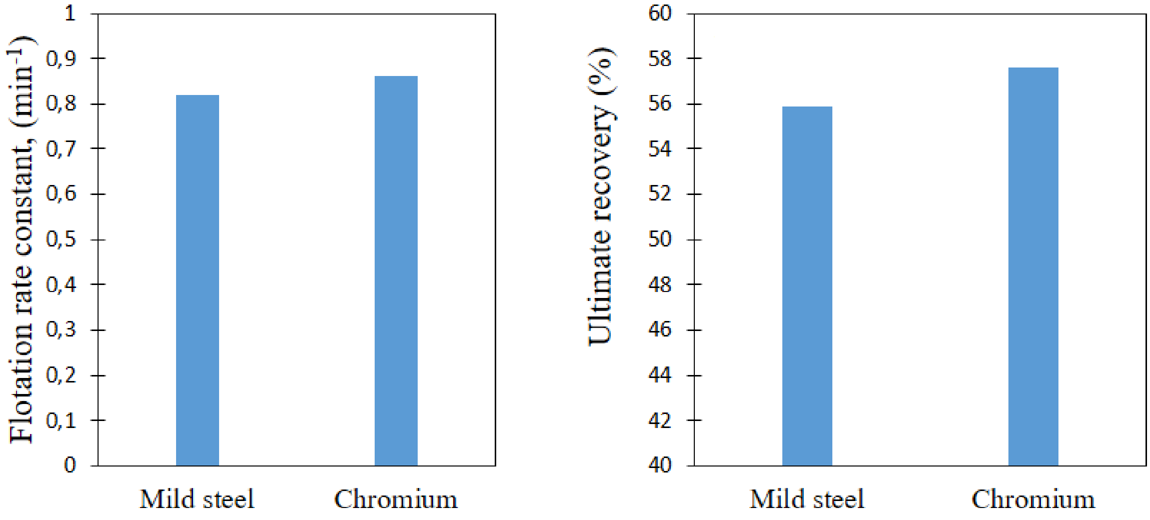


Figure 12. Effect of grinding chalcopyrite by different grinding media on A: Flotation rate constant and B: Ultimate recovery



(A) (B)

Figure 13. Effect of grinding chalcopyrite-pyrite by different grinding media on A: Flotation rate constant and B: Ultimate recovery

chalcopyrite-pyrite mixture recovery versus time was observed in Figure 8. Chromium grinding balls show the same pattern of behavior as mild steel grinding balls because of sophisticated galvanic interactions between chalcopyrite, pyrite, and the medium.

The evaluation of R∞ or k individually is complicated since variation in the flotation conditions may cause a change in both R∞ and k. That means a change in a condition may increase R∞, while, as unexpected, may decrease k. Thus, the comparison of either R∞ or k obtained under different galvanic conditions can be misleading. They should be evaluated at the same time and through a valid method. To this end, the modified rate constant (K_m) is introduced (Equation 13), which can be defined as the product of R∞ and k and better describes the flotation kinetics [19]. Xu defined K_m as the slope of the tangent of the recovery-time curve at zero flotation time [35].

$$K_m = R_\infty k \tag{13}$$

The role of K_m is to express the flotation constant rate and ultimate recovery simultaneously to determine the flotation kinetics more effectively. It is observed that in chalcopyrite-pyrite interaction, the chalcopyrite content plays an important role in the value of K_m (Figure 14). The more chalcopyrite content in the feed content leads to a higher value of K_m. Galvanic interactions through grinding chalcopyrite by ceramic and mild steel medium resulted in the maximum and minimum of K_m, respectively. The effect of the grinding medium is also indicated in the chalcopyrite flotation when the mixture of chalcopyrite-pyrite is ground by chromium or mild steel balls. The K_m in the flotation of chalcopyrite is higher for grinding chalcopyrite-pyrite mixture in grinding by chromium balls than products of mild steel balls. It is noticeable that the difference in the results of these two media is not significant, the same exactly its cumulative recovery-time (Figures 8 and 14), which is in good agreement with cumulative recovery-time charts (Figures 4-8). The more galvanic interactions, the lower K_m.

4- CONCLUSIONS

The effect of galvanic contact of- chalcopyrite-pyrite, chalcopyrite-different grinding balls, and chalcopyrite-pyrite- different grinding balls-, and also the impact of these galvanic interactions on the flotation kinetics were investigated. The resting potential analysis assessments indicated, as it was an obvious fact, that pyrite was nobler than chalcopyrite, chromium, and steel balls, respectively, and it was also nobler than chalcopyrite in the presence of xanthate as the

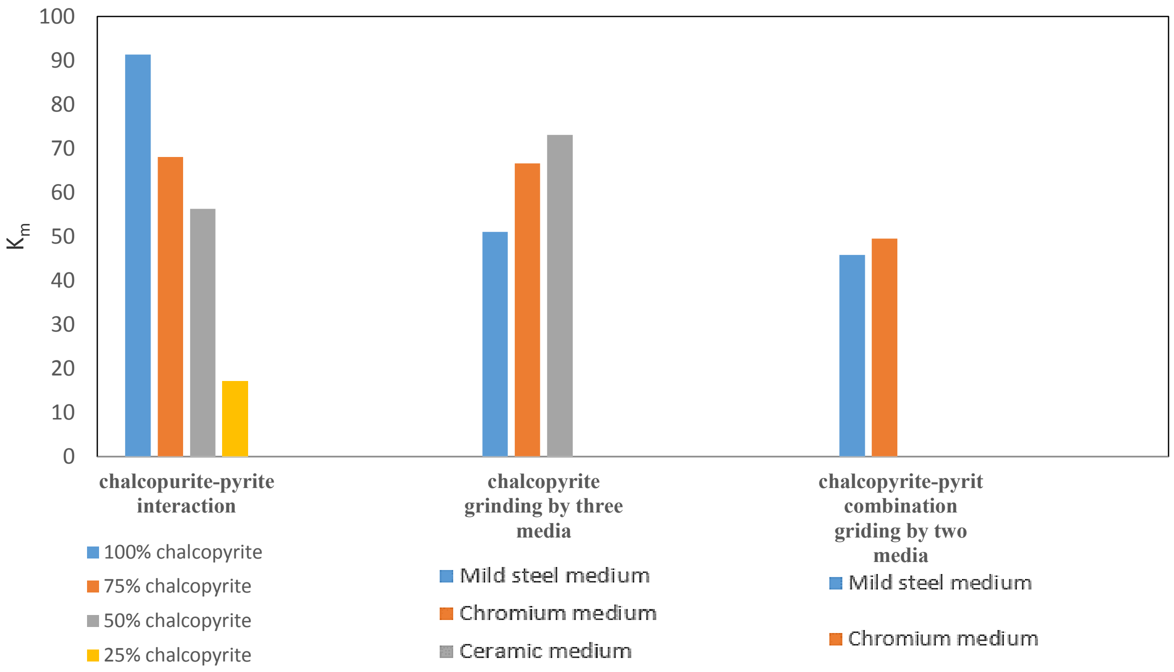


Figure 14. K_m **in different galvanic interactions**

main sulfide collector. Since pyrite had a higher resting potential, was the nobler, and showed lower electrochemical activity, during the process, it served as a cathode in galvanic coupling for other materials. Outcomes indicated that the flotation recovery of chalcopyrite decreased by increasing the pyrite content in the mixture. The EDTA technique studies suggested the oxidation of chalcopyrite and the activation of pyrite through the process. The flotation recovery of chalcopyrite increased when ceramic balls were used for grinding. Grinding by steel balls produced the highest iron oxidation species. While ceramic grinding balls produced the most and the least copper and oxidation species, respectively. However, grinding the mixture of chalcopyrite-pyrite by chromium and steel balls produced the same amount of flotation recovery. Results also indicated that the higher rest potential difference among chalcopyrite-pyrite-steel balls than chalcopyrite-pyrite-chromium balls caused more dissolution of the chalcopyrite surface. The evaluation of the first-order kinetics models suggested that the classic model had the most accurate fit to the experimental data of flotation recovery. By increasing chalcopyrite content in the mixture from 25 to 50%, the flotation rate constant increased. Afterward, by increasing from 50 to 75%, it decreased due to pyrite activation. While the ultimate recovery increased by increasing chalcopyrite content in the mixture. Grinding single chalcopyrite by chromium grinding balls produced a higher flotation rate constant than other grinding media, and grinding by ceramic balls produced the highest flotation recovery. Grinding the mixture of chalcopyrite-pyrite by chromium and steel balls resulted in the same pattern of flotation rate constant and ultimate recovery in agreement with the flotation recovery.

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علمی-پژوهشی

تاثیر برهمکنش‌های گالوانیکی بین محیط‌های آسیا و کانی‌های سولفیدی بر سینتیک فلوتاسیون کالکوپیریت

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چکیده

اگرچه جداسازی فلوتاسیون سولفیدها و برهمکنش‌های الکتروشیمیایی آنها در برخی تحقیقات بررسی شده است، اما مطالعه رابطه مستقیم بین سینتیک شناورسازی سولفیدها و برهمکنش‌های گالوانیکی آنها در طول فرآیند هنوز مورد بررسی قرار نگرفته است. برای پر کردن این شکاف، یک مطالعه گسترده برای بررسی اثر برهمکنش‌های گالوانیکی کالکوپیریت و پیریت بر سینتیک شناورسازی آنها هنگام خیس شدن، به وسیله سه نوع آسیاب مختلف (فولاد، کروم و سرامیک) انجام شد. ارزیابی نتایج آزمایش فلوتاسیون نشان داد که هیچ برهمکنش گالوانیکی بین گلوله‌های خردایش سرامیکی و کالکوپیریت وجود ندارد. فعل و انفعالات گالوانیکی بیشتر مربوط به آسیاب کالکوپیریت به وسیله گلوله‌های فولادی بود. آنالیزهای EDTA نشان داد که با افزایش میزان اکسی/هیدروکسی آهن و رسوب آنها بر روی سطح کالکوپیریت، بازیابی کالکوپیریت کاهش یافت. علاوه بر این، افزایش محتوای پیریت در فلوتاسیون کالکوپیریت باعث افزایش ثابت نرخ شناور و سپس کاهش آن شد. آزمایش‌های فلوتاسیون نشان داد که آسیاب کالکوپیریت با نوع محیط سرامیکی که دارای فعالیت الکتروشیمیایی پایین‌تری در مقایسه با سایر محیط‌های در نظر گرفته شده است، به ثابت نرخ شناور بالاتر و بازیابی نهایی منجر می‌شود. آسیاب کردن مخلوط کالکوپیریت و پیریت به وسیله محیط‌های فولادی یا کروم تقریبا همان اثر را روی ثابت نرخ شناورسازی و بازیابی نهایی نشان می‌دهد. به طور کلی نتایج آزمایش‌های انجام شده نشان داد که با افزایش اندرکنش گالوانیکی، بازیابی فلوتاسیون و سینتیک فلوتاسیون کاهش می‌یابد.

کلمات کلیدی

برهمکنش گالوانیکی، گلوله‌های خردایش، کالکوپیریت، پیریت، استخراج EDTA.

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