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EFFECTS OF EDTA ON SELECTIVE FLOTATION OF SULPHIDE MINERALS

Unwanted activation of sphalerite and pyrite by copper ions dissolved from copper minerals (mainly chalcopyrite) hampers flotation selectivity. Galvanic interaction between the sulphide minerals is considered as the main cause for such activation. Therefore, a complexing agent, EDTA, was used to prevent unwanted activation of sphalerite and pyrite by copper ions in the presence and absence of collector. In this study, unwanted activation of sphalerite and pyrite by copper ions and inhibition of this activation by using EDTA was investigated by using cyclic voltammetry and microflotation techniques. The influence of galvanic interaction between chalcopyrite-pyrite and chalcopyrite-sphalerite on flotation selectivity and the effect of EDTA were determined with microflotation tests performed with single mineral and mineral mixtures. The results pointed out dual effect of EDTA as preventing activation of sphalerite and pyrite by copper ions, but at the same time enhancing their collectorless flotation by removing the metal hydroxides from their surfaces.

Key words: flotation, sulphide minerals, activation, EDTA

INTRODUCTION

Oxidation of sulphide minerals is inevitable during processing in plant conditions. Every sulphide mineral is influenced at different degrees of oxidation by its chemical composition, crystal structure and most important its electrochemical reactivity (Ekmekçi et al. 2003). It is very well known that the flotation behaviour of a sulphide mineral, when mixed with other sulphide minerals in an ore, can be substantially different from that predicted by single and mixed mineral studies.

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The reasons suggested for this behaviour were attributed to galvanic interactions between sulphide minerals (Nakazawa and Iwasaki 1985; Yelloji Rao and Natarajan 1989; Ekmekçi and Demirel 1997). Unwanted activation and flotation of sphalerite in a complex lead/zinc ore or copper/zinc ore is an example to this type of behaviour. Therefore, determination of the degree of oxidation, surface species and their influence on flotation behaviour is necessary to develop processing strategies to minimize the negative effect of oxidation on flotation.

Clarke et al. (1995) has suggested some techniques to remove oxidation products, particularly metal hydroxides, from mineral surface. These techniques can be either chemical (dissolution by changing the pH, extraction by EDTA) or mechanical (sonification and attrition with quartz). Pulp pH can be adjusted to values outside the range of metal hydroxide formation and therefore mineral recovery can be improved. This method was found to prevent formation and precipitation of metal hydroxides rather than removing the previously formed oxidation products. Sonification is also considered as a potential method for selectively removing oxidation products from mineral surfaces. Preferential adsorption of metal ions dissolved from mineral surfaces on to quartz particles increased flotation performance significantly. However, it has been recognized by numerous workers (Shannon and Trahar 1986; Grano et al. 1988; Rumball and Richmond 1996) that EDTA has the ability to solubilize the oxidation products of sulphide minerals.

The negative effect of galvanic interaction in selective flotation, in enhancing the activation of pyrite by copper ions, has already been emphasized. This interference from metal ions can be prevented by the use of EDTA as a complexing agent to extract metal ions from the mineral surfaces and form complex compounds with the dissolved ions in the pulp. In this work, unwanted activation of sphalerite and pyrite by copper ions and inhibition of this activation by using EDTA was investigated by using cyclic voltammetry and microflotation techniques. The influence of galvanic interaction between chalcopyrite-pyrite and chalcopyrite-sphalerite on flotation selectivity and the effect of EDTA were determined by using mineral mixtures in microflotation scale.

MATERIALS AND METHODS

MINERALS

Natural samples of sulphide minerals of high purity were used in this study. Chalcopyrite and sphalerite were supplied by Wards Natural Science Establishment and pyrite crystals were obtained from Murgul (Artvin, Turkey) copper ore deposit.

Bulk analysis of the samples was performed by using Atomic Absorption Spectrometer at Mining Engineering Department of Hacettepe University. The results are given in Table 1. In addition to the chemical analysis, XRD analysis of the three mineral samples was also performed. The results confirmed purity of the samples (Ekmekçi et al. 2002).

Table 1. Results of bulk analysis of the natural mineral samples used in this study

Element (%)	Pyrite (FeS_2)	Chalcopyrite ($CuFeS_2$)	Sphalerite (ZnS)
Fe	44.67	29.14	1.22
Cu	0.16	35.28	0.065
Pb	0.016	0.006	0.57
Zn	0.004	0.028	60.68
S	54.13	33.30	33.61
Total	98.98	97.754	96.145

CYCLIC VOLTAMMETRY EXPERIMENTS

A conventional three-electrode system was used for electrochemical measurements in which a saturated calomel electrode, a platinum foil electrode with 1 cm^2 area and a mineral electrode were used as reference, counter and working electrodes respectively. The chalcopyrite and pyrite electrodes were prepared from highly mineralized massive specimens. A section of mineral with a rectangular cross-section was cut and mounted in a glass tube with an electrochemically inert epoxy resin. However, a different method was used to prepare sphalerite electrode due to its high resistivity compared to chalcopyrite and pyrite. Therefore, sphalerite was dry ground and the -100+150 μm fraction was mixed with carbon paste for electrode fabrication as proposed by Yoon and Chen (1996).The carbon paste was composed of graphite and conductive binder. The carbon paste was softened with ethyl ether and sphalerite particles were mixed at a ratio of 1/3 (mineral/paste). The homogenized mixture was then pressed into a glass tube and dried for one day for hardening. Therefore, current changes occurring at sphalerite surface were transferred with the aid of conductive carbon paste to a copper wire and then to a potentiostat.

Surface of the mineral electrodes was polished wet using 800-grit silicon carbide paper and then 1 μm diamond paste. After polishing the surface, the electrode was rinsed with distilled water and quickly transferred to the electrochemical cell. Depending on the experimental conditions $CuSO_4$ is first added and then EDTA into the electrochemical cell. The electrodes were conditioned for 5 minutes for each reagent addition and then scanning was started. All the experiments were performed in borate buffer solution of pH=9.2. Bank Elektronik Wenking PGS95 potentiostat/galvanostat was used for the cyclic voltammetry experiments. All the potentials reported in this paper have been converted to the standard hydrogen electrode (SHE).

MICROFLOTATION TESTS

Borate buffer solution of pH=9.2 was used in the microflotation tests. The tests were performed in a modified Hallimond tube. The mineral particles were dry ground and the fraction of -150+100 μm was used in the tests. Before conditioning, high purity (%99.99) nitrogen gas was intensively bubbled in the cell to remove dissolved oxygen. The same gas was also used for flotation. For each reagent addition the conditioning was taken as 2 minutes. Flotation was carried out for a total of 8 minutes with concentrate samples collected after 1, 3, 5 and 8 minutes of flotation. The flotation concentrates and the tail were filtered, dried and the mass of the mineral, and hence the recovery, was calculated for single mineral experiments. However, the concentrates were analyzed for Cu, Zn and Fe in the tests performed with mixtures of minerals. Calculation of the mass of mineral present in each fraction could then be made. Conditioning was performed in the following order depending on the number of reagents added; copper ions, EDTA and KEX.

RESULTS AND DISCUSSION

VOLTAMMETRY

SPHALERITE (ZnS)

Voltammograms of sphalerite taken with and without Cu^{2+} and EDTA at pH=9.2 are given in Figure 1. The scanning was commenced from the cathodic region and performed between -500 mV and +500 mV. Figure 1 shows that there is no significant electrochemical activity in this potential range in the absence of reagents. However, as the sphalerite electrode was treated with 10^{-4} M $CuSO_4$, an anodic peak appeared at about 300 mV. This anodic peak corresponds to oxidation of CuS type compounds formed as a result of copper activation.

It is very well known that majority of copper ions added into alkaline solutions precipitate in the form of $Cu(OH)_2$ (Fuerstenau and Fuerstenau 1982). Copper hydroxide can rapidly adsorb on sphalerite surface. However, copper sulphide may form in the following stages due to low stability of $Cu(OH)_2$. At the end of activation process, sphalerite surface may be transformed into (Zn, Cu)S form. Surface analysis of copper activated sphalerite in alkaline solution by XPS has shown CuS like surface (Clarke 1997). Therefore, the anodic peak observed at about 300 mV may correspond to oxidation of CuS-like species to copper oxide and polysulphides.

After copper activation, sphalerite electrode was conditioned in the presence of 10^{-3} M EDTA to remove the adsorbed copper ions from the surface (Figure 1). The anodic peak remained in the presence of EDTA under stirred conditions, confirming formation of copper sulphide rather than copper hydroxide as an end product even at pH 9.2.
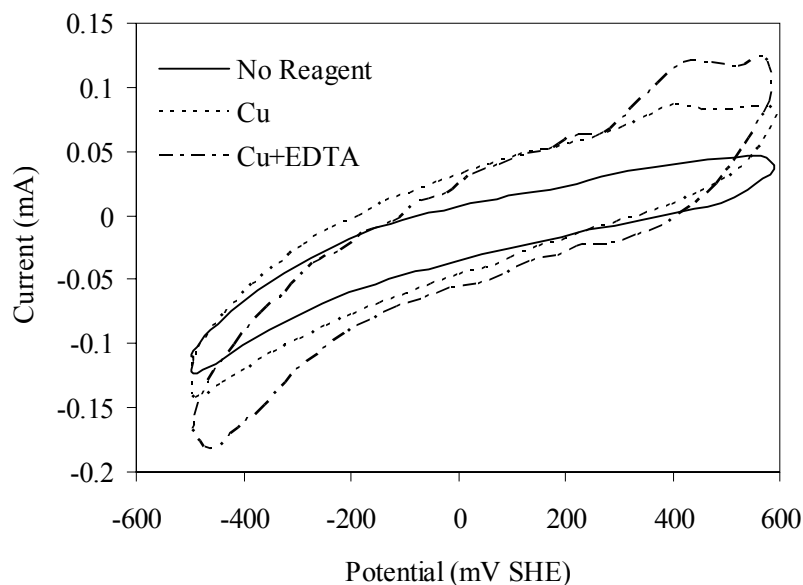
Fig. 1. Voltammograms of sphalerite with no reagent (——), 10^{-4} M Cu^{2+} (- - - - - ·)
and 10^{-4} M Cu^{2+} and 10^{-3} M EDTA (— · — · —) at pH=9.2. (Scan rate=50 mV/s)

PYRITE (FeS$_2$)

Voltammograms of pyrite taken with and without Cu^{2+} and EDTA are illustrated in Figure 2. One anodic and one cathodic peak were observed in the potential range of -500 mV and +500 mV. The anodic peak, having the maximum current at about 0 mV, corresponds to oxidation of ferrous hydroxide to ferric hydroxide form (reaction 2). This process is a reversible process and the cathodic peak corresponds to reduction of ferric hydroxide to ferrous hydroxide in the reverse direction of reaction 2.

$$Fe(OH)_2 + OH^- \leftrightarrow Fe(OH)_3 + e^- \tag{1}$$
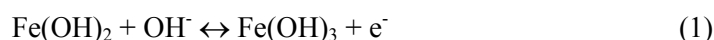
Reversible potential of this reaction was calculated as 8.3 mV for pH 9.2, which is fairly close value to that observed in Figure 2.

Activation of pyrite by copper ions was also investigated using cyclic voltammetry. The voltammogram of pyrite taken in the presence of 10^{-4} M Cu^{2+} is given in Figure 2. It appears that in addition to the anodic and cathodic peaks showing oxidation/reduction of iron hydroxide, a new anodic appeared at about 300 mV after copper activation. It should be noted that this anodic peak appeared at the same potential to that observed with sphalerite after copper activation (Figure 1), corresponding to oxidation of CuS (reaction 1).
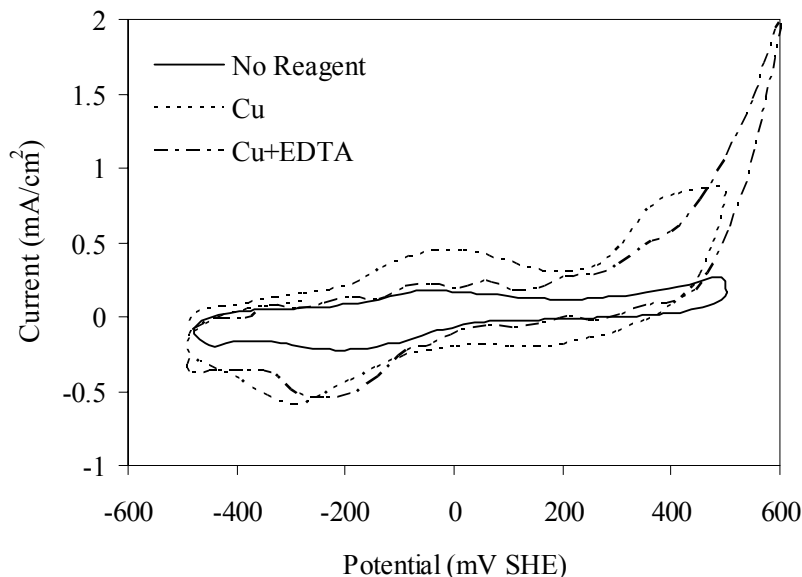
Fig. 2. Voltammograms of pyrite with no reagent (———), 10^{-4} M Cu^{2+} (- - - - -)
and 10^{-4} M Cu^{2+} and 10^{-3} M EDTA (— · — · —) at pH=9.2. (Scan rate=50 mV/s)

Voltammogram of copper activated pyrite in the presence of EDTA showed that the anodic peak showing oxidation of ferrous iron to ferric iron almost disappeared. It is well known that EDTA can easily remove the iron hydroxide species and therefore disappearance of the anodic peak at about 0 mV was anticipated. In addition to that current density of the second anodic peak, observed after copper activation, decreased substantially, in spite of the fact that CuS has higher stability than Cu-EDTA complex and therefore should not be affected by addition of EDTA. This may be related to kinetics of copper activation of pyrite, which may be slower than with sphalerite. Therefore, a large portion of copper ions might not replace iron in pyrite and still remained as $Cu(OH)_2$ in the vicinity of the surface. Addition of EDTA may remove these species and resulted in considerable decrease in the current intensity of the corresponding anodic peak.

CHALCOPYRITE ($CuFeS_2$)

Voltammograms of chalcopyrite in the absence and presence of 10^{-4} and 10^{-3} M EDTA is illustrated in Figure 3. There were no changes in the shape of chalcopyrite voltammograms in spite of excess EDTA concentration. This result showed that EDTA did not affect chalcopyrite surface, as the anodic peak observed at about 300 mV corresponds to oxidation of chalcopyrite producing CuS at the surface.
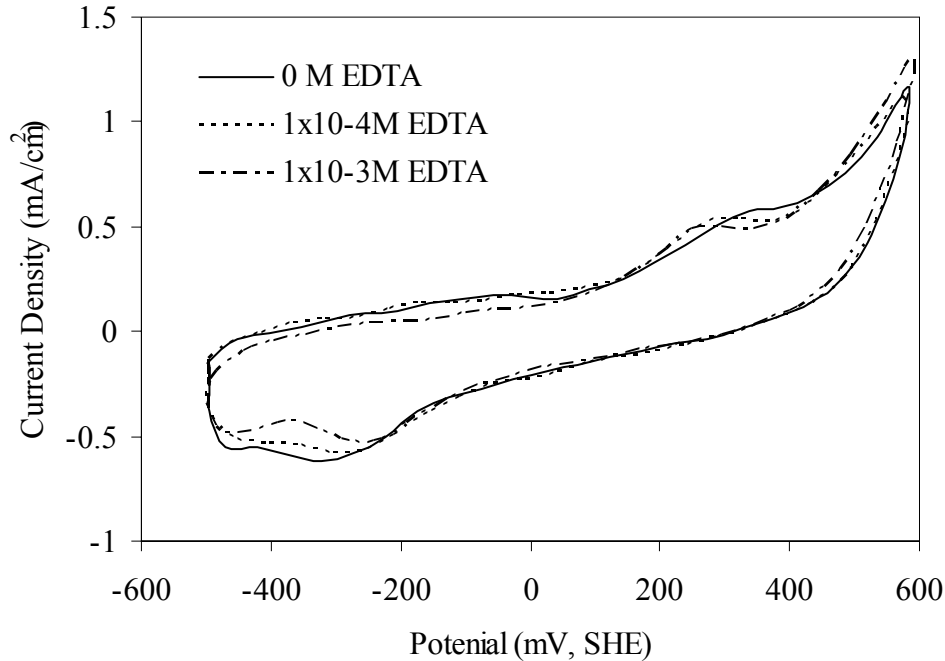
Fig. 3. Voltammograms of chalcopyrite with no reagent (———), 10^{-4} M EDTA (- - - -)
and 10^{-3} M EDTA (— · — · —) at pH=9.2. (Scan rate=50 mV/s)

MICROFLOTATION EXPERIMENTS

Microflotation experiments give valuable information about the nature of the species, as hydrophobic or hydrophilic, which is very important for flotation. In this study flotation behaviour of the minerals were tested as single mineral and mineral mixtures (chalcopyrite-sphalerite and chalcopyrite-pyrite) in the absence and presence of ethyl xanthate (KEX). Therefore, effects of galvanic interaction and EDTA on selective flotation were investigated.

SINGLE MINERAL

SPHALERITE

Results of sphalerite flotation in the absence and presence of copper and EDTA are shown in Figure 4. Flotation recoveries of sphalerite in the absence and presence of copper ions were very low due to formation of hydrophilic $Zn(OH)_2$ and $Cu(OH)_2$ species respectively at pH 9.2. However, addition of EDTA after copper activation increased the recovery drastically from about 10 % up to 40 %.
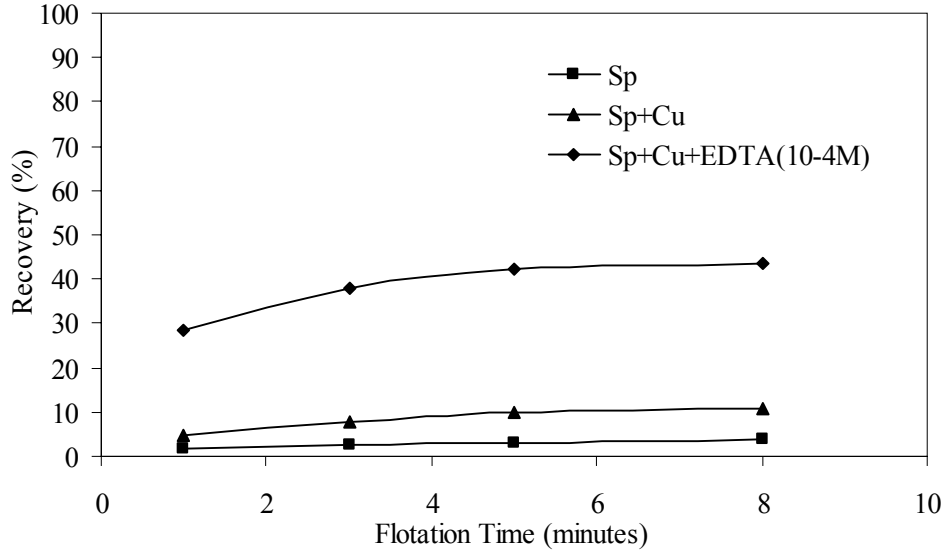
Fig. 4. Collectorless flotation of sphalerite in the absence and presence of copper and EDTA at pH 9.2

The increase in the recovery after EDTA addition was attributed to removal of copper and zinc hydroxide species and exposure of elemental sulphur or polysulphides, which made sphalerite hydrophobic to some extent. As it has been mentioned above, the copper ions are found in the form of $Cu(OH)_2$ in alkaline solutions. These species can be first physically adsorbed or precipitate on to sphalerite surface and then proceed with electrochemical adsorption through reduction of Cu^{2+} to Cu^+ (reaction 2). As a result of that sulphide (S^{2-}) ions at the surface of sphalerite are oxidized into elemental sulphur or polysulphides (reaction 3) (Finkelstein 1997). Therefore, after removal of metal oxides by EDTA from the surface, a hydrophobic surface exposed and the recovery increased.

$$Cu^{2+} + e^- \rightarrow Cu^+ \tag{2}$$

$$\tfrac{1}{2}S^{2-} \rightarrow \tfrac{1}{2}S° + e^- \tag{3}$$

Addition of 10^{-4} M KEX did not increase the recovery of sphalerite in the absence of copper ions (Figure 5). As it is very well known that solubility of Zn-EX (4.9×10^{-9}) is considerably higher than $Zn(OH)_2$ (1.62×10^{-17}) and therefore xanthate can not adsorb on sphalerite. Therefore, copper activation is essential for flotation of sphalerite and as it is shown in Figure 1, sphalerite can easily be activated by copper ions even in alkaline solutions. Addition of KEX resulted in formation of strongly hydrophobic CuEX, and thus very high recoveries.

Addition of 10^{-4} M EDTA did not affect the recovery. This is in agreement with the cyclic voltammetry results (Figure 1) showing no effect of EDTA addition to copper activated sphalerite. However, as EDTA concentration was increased to 10^{-3} M, the recovery and flotation rate decreased significantly. The decrease in the recovery after excess EDTA addition may be due to break down of the collector species adsorbed at the surface and/or precipitation of EDTA complexes at the surface.
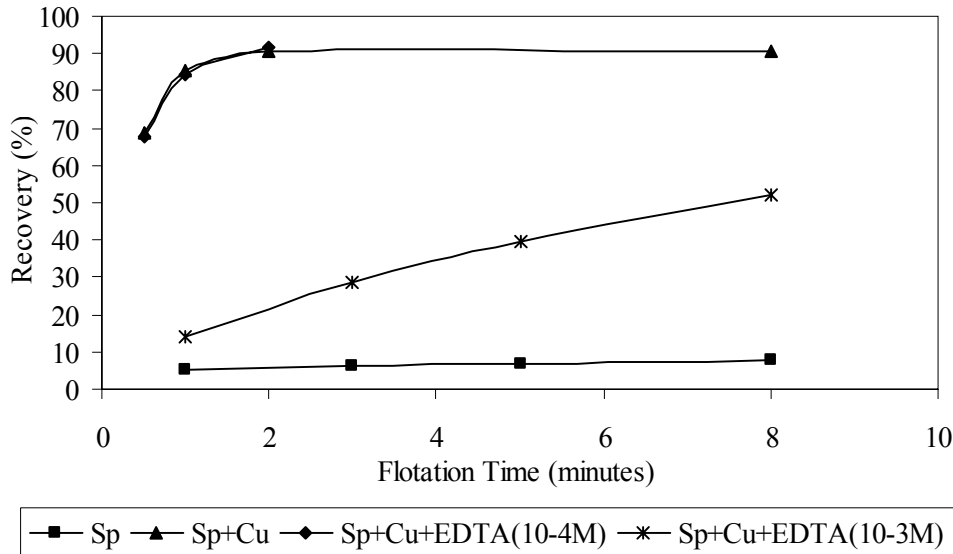

Fig. 5. Flotation of sphalerite with 10^{-4} M KEX in the absence and presence of copper and EDTA

PYRITE

Collectorless flotation behavior of pyrite in the absence and presence of copper ions and EDTA is illustrated in Figure 6. Collectorless flotation of pyrite is very low due to formation of hydrophilic iron oxide/hydroxide species in alkaline solution. Addition of copper ions increased the recovery slightly due to formation of elemental sulphur or polysulphides to some extent as also observed in the case of sphalerite (Figure 4). Addition of EDTA, however, increased the recovery considerably due to removal of hydrophilic ferric hydroxide species and exposure of sulphur rich hydrophobic layer at the surface.

Addition of KEX did not affect pyrite recovery significantly (Figure 7). Flotation of pyrite strongly depends on formation of dixanthogen which also depends on the presence of oxygen in the pulp. However, the flotation experiments were performed in the absence of oxygen by bubbling nitrogen gas prior to flotation. Therefore, absence of oxygen in the solution might inhibit formation of dixanthogen and resulted in low recovery of pyrite.

Z. Ekmekçi, A. Aslan, H. Hassoy

Addition of copper ions increased the recovery drastically up to 75 %. This was apparently due to activation of pyrite by copper ions, as shown by cyclic voltammetry in Figure 2, and formation of CuEX species upon addition of collector. However, addition of EDTA after copper activation could remove the adsorbed copper ions largely as shown by voltammetry experiments. Therefore, addition of EDTA before xanthate addition removed most of the pre-adsorbed copper ions and therefore prevented formation of CuEX at pyrite surface. In spite of that the recovery was still around 50 %. It was clear that addition of EDTA inhibited collector adsorption, but at the same time removed metal hydroxide species and resulted in exposure of sulphur rich hydrophobic surface, as observed with collectorless flotation (Figure 6).
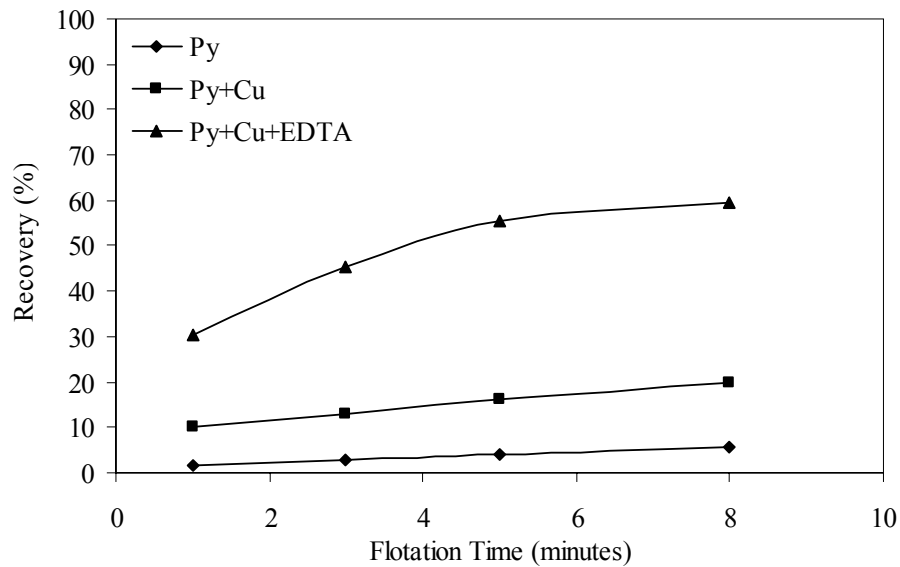

Fig. 6. Collectorless flotation of pyrite in the absence and presence of copper ions and EDTA

CHALCOPYRITE

In this section, only influence of EDTA on collectorless flotation of chalcopyrite was discussed since chalcopyrite was not affected by EDTA at all in the presence of KEX. Addition of EDTA increased the collectorless flotation recovery from about 30 % up to about 90 % (Figure 8). Electrochemical experiments showed that oxidation of chalcopyrite in alkaline solutions resulted in formation of ferric hydroxide at moderately oxidizing potentials and copper hydroxide at higher potentials. These hydrophilic metal hydroxide species cover the mineral surface and prevent its collectorless flotation. Addition of EDTA removed all of these metal hydroxide species and brought about exposure of a sulphur rich hydrophobic layer, as observed with collectorless flotation of both sphalerite and pyrite.
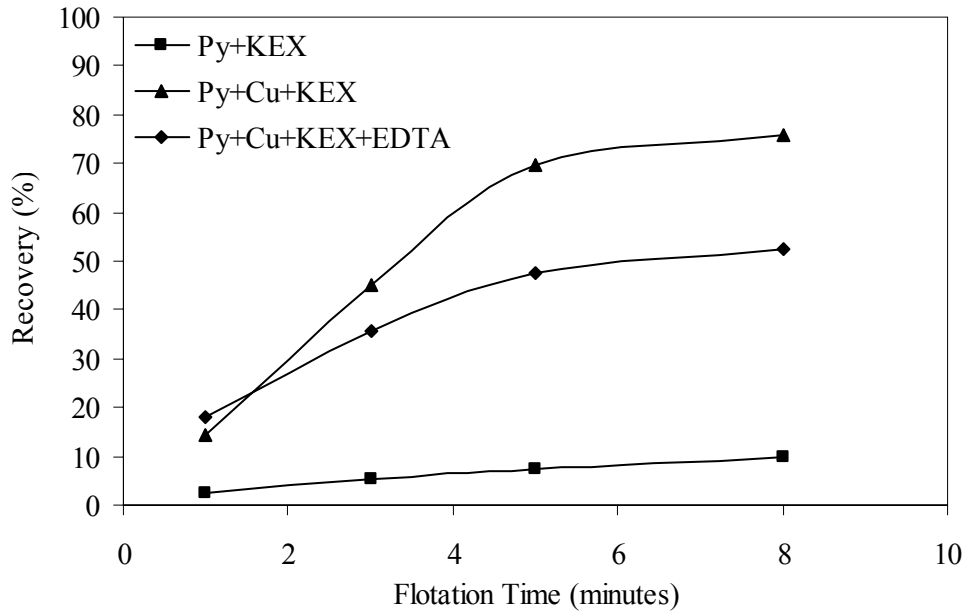
Fig. 7. Flotation of pyrite with 10^{-4} M KEX in the absence and presence of copper ions and EDTA
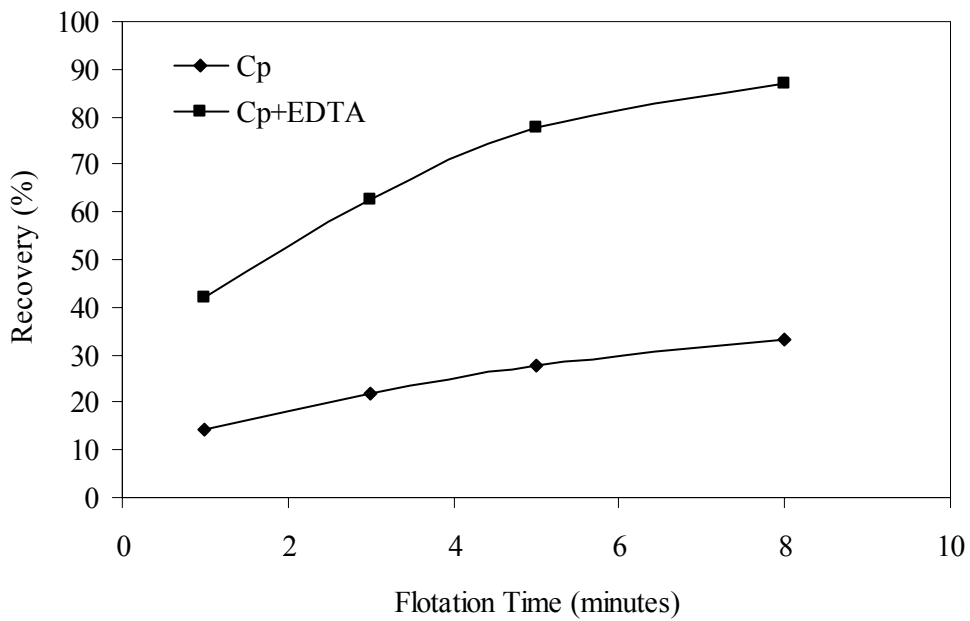

Fig. 8. Collectorless flotation of chalcopyrite in the absence and presence of 10^{-4} M EDTA at pH 9.2

MINERAL MIXTURES

Effects of galvanic interaction on activation and flotation of sphalerite and pyrite were investigated with microflotation experiments performed using chalcopyrite-sphalerite and chalcopyrite-pyrite mineral mixtures. It is very well known that galvanic interaction between these minerals cause dissolution of copper ions from chalcopyrite and thus unwanted activation of sphalerite and pyrite by these copper ions, which deteriorates the selectivity. Therefore, EDTA was tested as a complexing agent to prevent activation of sphalerite and pyrite in these experiments.

The results of collectorless flotation of chalcopyrite and sphalerite in the absence and presence of EDTA are illustrated in Figure 9. Presence of chalcopyrite did not affect sphalerite recovery in the absence of EDTA. However, as EDTA removed the metal hydroxide species from surface of both minerals, their recovery increased considerably, as it was also observed with single mineral experiments.

Addition of KEX increased flotation recovery of sphalerite in the mineral mixture (Figure 10). This was attributed to activation of sphalerite by copper ions dissolved from chalcopyrite as a result of galvanic interaction. However, addition of EDTA decreased the recovery in the order of 10 %. The final sphalerite recovery in the presence of EDTA was still about 30 %. EDTA was considered to have dual effect as preventing activation of sphalerite by copper ions, but at the same time enhancing its collectorless flotation by removing the metal hydroxides from the surface. Therefore, selective flotation could not be achieved at satisfactory level in the existing experimental conditions. The influence of pH and particularly EDTA concentration on selectivity must be investigated in detail.
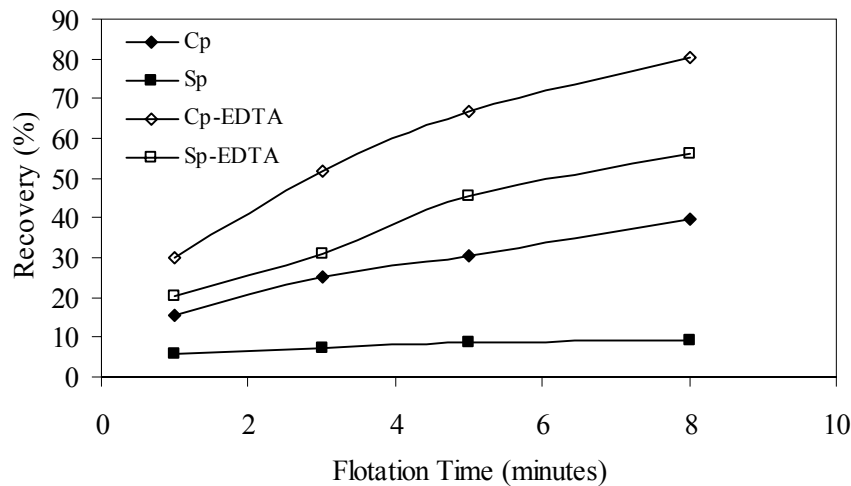

Fig. 9. Collectorless flotation of chalcopyrite and sphalerite in mineral mixture in the absence and presence of EDTA
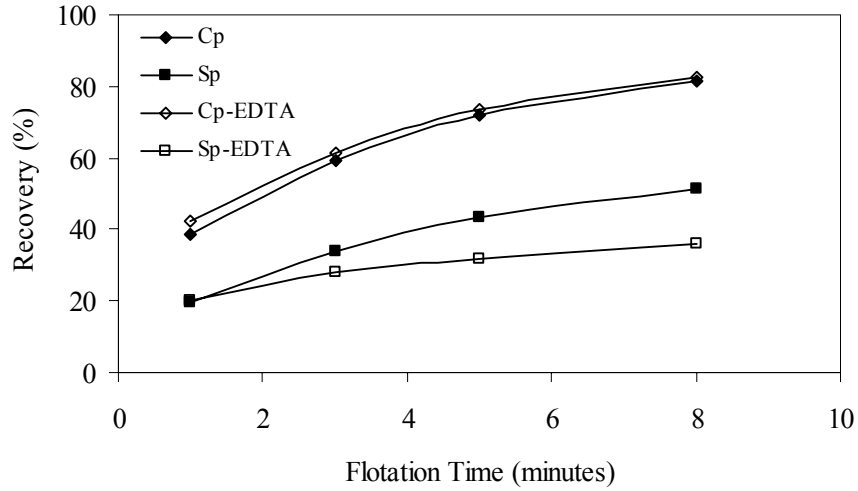
Fig. 10. Flotation of chalcopyrite and sphalerite in mineral mixture with 10^{-4} M KEX in the absence and presence of EDTA

Similar flotation behaviour was also observed with chalcopyrite-pyrite mixture in the absence and presence of KEX (Figures 11 and 12). Collectorless flotation of both chalcopyrite and pyrite in mineral mixture was not significantly different from that in single mineral experiments. However, addition of EDTA removed the metal hydroxide compounds for their surfaces and increased recovery of both minerals.
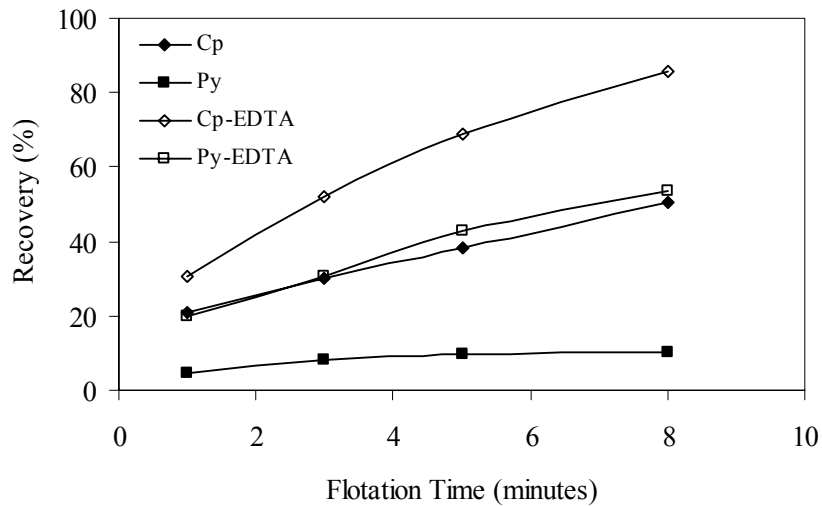

Fig. 11. Collectorless flotation of chalcopyrite and pyrite in mineral mixture in the absence and presence of EDTA

Recovery of pyrite in the presence of KEX increased to about 40 % due to galvanic interaction between chalcopyrite and pyrite. Dissolution of copper ions enhanced due to galvanic interaction. The dissolved copper ions may adsorb/precipitate on pyrite and form CuEX compounds after KEX addition. Addition of EDTA removed the copper ions from pyrite surface and prevent formation of CuEX, but it also removed the metal hydroxides and led to collectorless flotation of pyrite. Flotation of chalcopyrite was not affected by addition of EDTA in the existing experimental conditions.
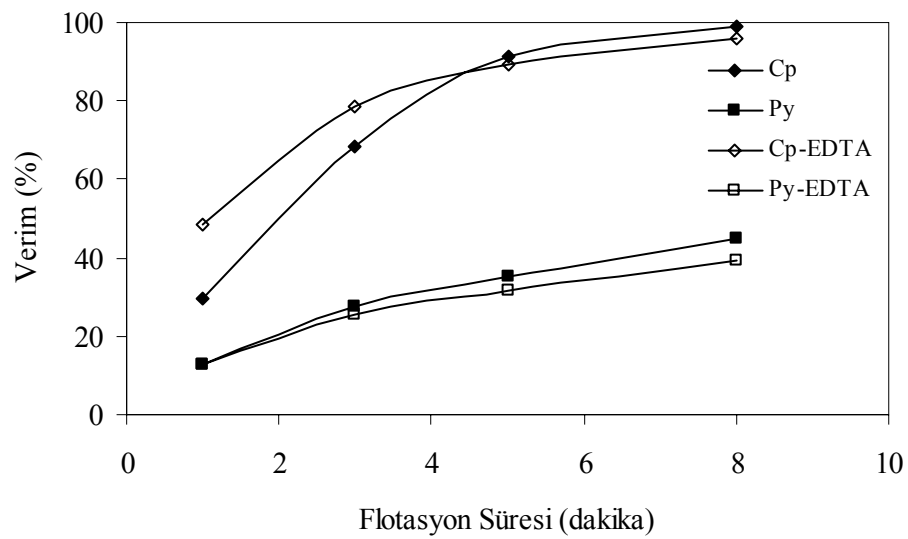

Fig. 12. Flotation of chalcopyrite and pyrite in mineral mixture with 10^{-4} M KEX in the absence and presence of EDTA

CONCLUSIONS

Unwanted activation of sphalerite and pyrite by copper ions dissolved from copper minerals (mainly chalcopyrite) hampers flotation selectivity. Galvanic interaction between the sulphide minerals is considered as the main cause for such activation. Therefore, a complexing agent, EDTA, was used to prevent unwanted activation of sphalerite and pyrite by copper ions in the presence and absence of collector.

Cyclic voltammetry experiments showed that sphalerite and pyrite can be activated by copper ions at pH 9.2, forming CuS like compounds at the surface. Addition of EDTA did not affect the newly formed CuS layer with sphalerite, but decrease the current intensity with pyrite. The activated copper layer was weakly adsorbed onto pyrite surface, presumably due to slower adsorption kinetics of copper on pyrite compare to sphalerite.

The results of collectorless flotation revealed that flotation of chalcopyrite, pyrite and sphalerite was significantly affected by the presence of metal hydroxide at their surface. The use of EDTA removed these hydrophilic metal hydroxide species thereby exposing a sulphur rich hydrophobic surface. Similar effects were observed with both single mineral and mineral mixture experiments. This effect increased the overall recovery and did not improve the selectivity.

The flotation experiments in the presence of collector showed that galvanic interaction resulted in unwanted activation of both sphalerite and pyrite by copper ions, thereby resulting in formation of CuEX layer at their surface. This situation increased their recovery and hampers the flotation selectivity. EDTA was used to prevent copper activation. However, the flotation recovery of both sphalerite and pyrite remained almost the same in the presence and absence of EDTA. EDTA was considered to have dual effect as preventing activation of sphalerite and pyrite by copper ions, but at the same time enhancing their collectorless flotation by removing the metal hydroxides from their surfaces.

ACKNOWLEDGEMENT

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Proces aktywacji sfalerytu i pirytu przez jony miedzi, znajdujące się w roztworze, ma niekorzystny wpływ na selektywność procesu flotacji. Procesy galwaniczne są rozpatrywane jako główna przyczyna powodująca aktywację. Odczynnik kompleksujący- EDTA- został użyty w celu przeciwdziałaniu aktywacji sfalerytu i pirytu. Badania z wykorzystaniem EDTA prowadzone były bez i z udziałem kolektora. Metody cyklovoltamperometrii i mikroflotacji zostały wykorzystanie dla zbadania efektów wynikających z dodania EDTA. Dla układów chalkopiryt-sfaleryt i chalkopiryt-piryt określono wpływ EDTA na zjawiska galwaniczne i na selektywność flotacji. Otrzymane wyniki wskazują, ze dodanie EDTA zapobiega aktywacji sfalerytu i pirytu jonami miedzi, a także podwyższa bezkolektorową flotację tych minerałów przez usunięcie wodorotlenków metali z ich powierzchni.