Effect of pH on Pulp Potential and Sulphide Mineral Flotation

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Abstract

Control of pH is one of the most widely applied methods for the modulation of mineral flotation. In this study the effect of pH on potential in solution and sulphur minerals flotation is discussed with various electrodes. The electrodes were platinum, gold, chalcopyrite, pyrite and galena. In solution, potentials were linearly dependent on pH with a different slope for each electrode. Chalcopyrite, pyrite, sphalerite and galena minerals flotation tests were performed in a microflotation cell. Xanthates were used as a collector and their carbon chain length was tested at different pH values. As expected, pH has a significant effect on flotation and pulp potential. The effect of pH on sulphide flotation examined paying particular attention to the pulp potentials involved.

Key words: pH, Flotation, Electrochemical potential, Electrode, Sulphide minerals.

Introduction

The potential difference of a mineral-solution interface, which is indicated by pulp potential, has been shown to be closely related to the floatabilities of sulphide minerals. A good amount of literature is available on the subject (Natarjan and Iwasaki, 1973; Gardner and Woods, 1979; Hoyack and Raghavan, 1987; Woods, 1976; Janetski *et al.*, 1977; Kocabağ *et al.*, 1990a; Fuerstenau *et al.*, 1968; Maouf *et al.*, 1986; Göktepe and Williams 1995; Rand and Woods, 1983). It was reported that minerals can be made to float or sink alternately by changing the oxidising and reducing conditions in pulp conditions and the measured potentials determine whether or not the mineral will float. However, the quoted potential range for good flotation varies significantly and conflicting observations are present in the literature. Therefore explanations for the phenomena are still open to argument.

In order to monitor redox properties, a platinum electrode is usually used as an indicator electrode and placed in the solution, because it has high resistance to corrosion. The measured potential in flota-tion systems is the mixed potential and it is somewhere between the potential of the minerals and potential of the solution and different electrode materials can yield different Eh values in the same solution (Labonte and Finch, 1988). Labonte and Finch (1988), Rand and Woods (1983) and Woods (1976) reported that from the basic principles of flotation, the desired potential to be monitered would be the mineral potential, not the solution. This suggests that an electrode constructed from the mineral being concentrated should be the most appropiate for potential measurements rather than noble metal electrodes. Therefore, in the present study, different mineral and noble metal electrodes were immersed in the same system to determine the best sensing electrode for the electrochemical measurements in flotation of sulphide minerals. Factors such as the purity of the solution, the type of electrode used, and the history of the indicator electrodes were reported to have effects on the measurements of Eh values (Natarajan and Iwasaki, 1970).

Studies on the measurement of potential as a function pH have been performed in pure solution by Natarajan and Iwasaki (1972), Gebhart and Shedd

(1988), Ross and Van Deventer (1985) and Ahmed (1978). It was reported that the Eh/pH response of mineral and noble metal electrodes may point to certain implications of their surface conditions, and the flotation and leaching behaviours of sulphide minerals under aerated conditions may be inferred with respect to Eh/pH response.

The measurement of pulp potential in conjunction with the traditional pH measurements has also become more widespread in the industrial flotation circuit in recent years. Collection of Eh-pH data for an operating process may decrease reagent additions, and provide useful additional and helpful information for solving an operational problem for flotation plants (Johnson *et al.*, 1988). Eh/pH measurements were examined in relation to the flotation for pyrrhotite by Natarajan and Iwasaki (1973) and Eh was found to be a good indicator of the flotability of minerals.

In the present study, the most common four sulphide minerals are considered for Eh/pH relationship in the flotation process in a systematic manner.

Experimental

The microflotation cell was manufactured from a 400 ml Pyrex beaker, by forming two lips on either side and fixing four equally spaced baffles around the perimeter. A Perspex holder which could accomodate four electrodes and a gas distribution tube with a sintered end to provide a stream of fine bubbles was fixed above the cell. A glass cone was bonded to the end of the tube to provide a homogeneous gas

bubble distribution and to improve solid circulation. The tube was fed compressed gas for all tests via a flowmeter and was maintained at the same level in the beaker. A Teflon coated magnetic bar which was flatted on one surface was used to agitate the pulp and a hot plate was used to keep the temperature at $25 \pm 1°C$.

The potential and pH were measured using Corning Eh/pH meters (model 240). All potential measurements were against the Ag/AgCl reference electrode. Figure 1 shows the whole experimental apparatus.

Electrode Manufacture

The important potential which influences mineral flotation is that at its interface with the surrounding liquid. For this reason a range of mineral electrodes was manufactured for this study. Figure 2 shows the mineral electrode used for the experimentation. They were fabricated using pure pyrite, chalcopyrite and galena species. However, it was impossible to make a sphalerite electrode in this way because of sphalerite's poor conductivity. As shown in Figure 2 a brass button connector was bonded to the surface of highly pure specimen of the mineral under study by applying "Electrodag", a highly conductive paint. Then the sample was mounted using transoptic powder and ground and polished to adequate surfaces, free from scratches and with a minimum of relief. This required a range of abrasive and diamond pastes using standard techniques for preperation of samples for ore microscopy. After encapsulation, the
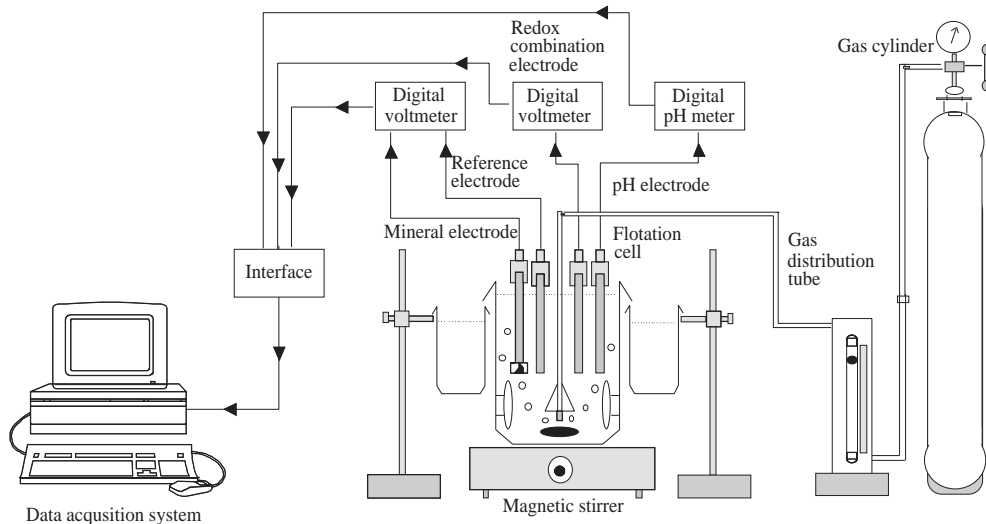

Figure 1. Experimental apparatus for microflotation experiment.

back of the sample was drilled and a 3 mm diameter brass rod screwed into the threaded part of the connector. The brass rod was then sleeved with a Perspex tube and adhesive was applied to seal both the top and bottom of the tube. An electrical connection was soldered to the top of the brass rod for a lead connection to the voltmeter.
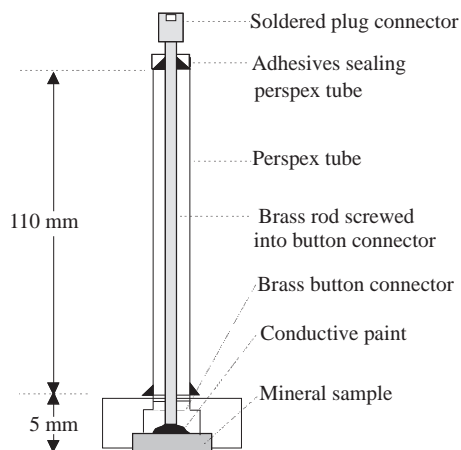

Figure 2. Schematic diagram of mineral electrode.

Reagents

Double distilled water was used for all microflotation tests. All solutions were prepared daily using commercial grade reagents. All collectors and the frother were kindly supplied by Cyanamid Ltd.

The strength of the stock solution was 0.1% for the collectors. The frother (Aerofroth 65) was also added pure in 25 μl amounts. Graduated microsyringes were used for each reagent with new needles for each. The microsyringes were cleaned with acetone every day after they were used.

Highly pure nitrogen (99.99%) was used as flotation gas. The compressed gas was supplied at a fixed rate from a cylinder.

Sample

In the pure mineral study four samples were used: sphalerite, pyrite, chalcopyrite and galena. The pure sulphide samples used were obtained from Gregory, Buttley & Lloyd, Mineralogist & Geologist, in London.

For the quantitative determination of the major elements present in the sample for microflotation, XRF analyses were performed. The results are shown in Table 1.

Table 1. XRF analyses of sulphide minerals used.

Sample	Metal %			
	Cu	Pb	Zn	Fe
Chalcopyrite	23.20	0.68	1.62	23.24
Pyrite	0.05	-	0.07	40.52
Sphalerite	0.14	-	66.59	7.84
Galena	0.07	89.90	0.10	2.23

XRF results show that except chalcopyrite and pyrite the other samples were highly pure; the major contaminating elements within the chalcopyrite sample were zinc and lead. The measured lead content of galena was higher than the theoretical percentage (89.90%), (Table 1).

Sample Preparation

Approximately 4 kg amounts of mineral samples were crushed to minus 10 mm with a porcelain mortar and pestle to prevent any metallic contamination common from mechanical crushers and grinding devices. Then the samples were homogenised and sub-sampled into 50 g lots in sealed bags and stored in a freezer maintained at -18°C until required for testing. This minimised any surface contamination or oxidation. Immediately prior to flotation, 10 g of sample was crushed to minus 0.5 mm with a porcelain mortar and pestle and wet ground with double distilled water in a micronising mill. The samples were ground for the required size fraction d_{80} of approximately 63 μm.

Standard Procedure for Microflotation Studies

After the required size was obtained by milling in the micronising mill the sample was transfered to the microflotation cell, and filled with 330 ml of double distilled water. All electrodes were washed with distilled water before each test. pH was measured to an accuracy of approximately ±0.01 units by Corning Eh/pH meters (model 240). The instrument was regularly checked with standard buffer solutions at pH 4, 7 and 10.

The data acquisition system was run until completion of the flotation to measure potential and pH every 15 seconds. The magnetic stirrer was set at a fixed value, 4, on the instrument dial. One minute conditioning was given for all collectors and frothers. For other added inorganic electrolytes 2 min were

given for conditioning. Nitrogen as flotation gas was introduced at 1 l/min by the gas distribution tube into the microflotation cell. The flotation tests were carried out for 4 min. Froth was removed by scraping manually and addition of double distilled water. Floated and unfloated products were then filtered, dried and weighed.

Results and Discussion

In solutions

Minerals and noble metal electrodes were periodically checked in double distilled water at natural pH to study their stabilities. The electrodes were stable within ±6mV with a 95% confidence level. Average values of the potentials as a function of time at natural pH are given in Figure 3. As expected, different minerals and noble metals gave different potentials. The order of electrode potential is platinum > gold > pyrite > chalcopyrite > galena. This follows the same order of minerals electrochemical activity. From noble to active is pyrite, chalcopyrite, galena and sphalerite. The activity of galena for oxygen reduction is less than that of the noble metals and chalcopyrite and pyrite. Lower Eh values were observed with galena by Rand and Woods (1984) as well. Although platinum and gold electrodes are both noble metal electrodes they showed 150-200 mV difference for the whole pH range. This was explained by a higher electrocatalytic activity of platinum for oxygen reduction when compared to gold by Rand and Woods (1983).
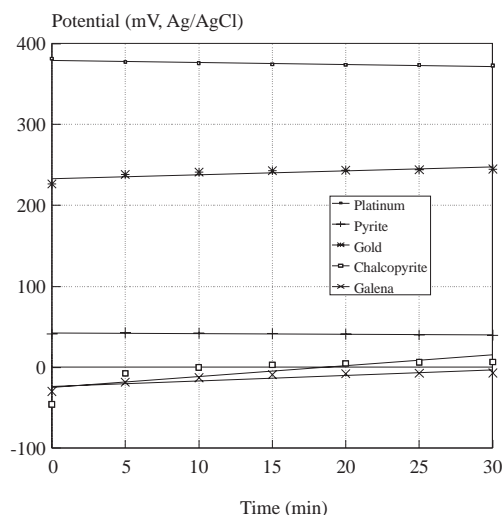

Figure 3. Potential against time for various electrode in double distilled water.

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Then pH was varied in double distilled water and electrochemical potential was measured with platinum, gold, chalcopyrite, pyrite and galena electrodes against pH variation. As shown in Figure 4, except for galena, potentials were linearly dependent on pH with a slope corresponding to 43.3 mV/pH for the platinum electrode, 32 mV/pH for gold, 13.8 mV/pH for chalcopyrite and 26 mV/pH for pyrite. Again except for galena all electrode potentials were get close to each other in the alkaline region. This is because of their passivation due to the formation of oxides and hydroxides (Natarajan and Iwasaki, 1972a). Ross and Van Deventer (1985), and Ahmed (1978) also found a linear relationship between potential and pH for chalcopyrite and pyrite; however, no such linear relationship between potential and pH existed for galena in solution in their study either. They reported that potential was stable around -25 mV (Ag/AgCl) between pH 3 to 8 and decreased sharply as pH increased to -105 mV(Ag/AgCl). In the present study potential was around -10 mV at pH up to 7, then decreased as pH increased and at pH 13 reached a value of –300 mV (Ag/AgCl) (Figure 4). This behaviour of galena electrodes was explained by Toperi and Tolun (1969) as follows: at higher pH values the dissolution of the passivating film increases the rate of anodic oxidation and the slope of the curves becomes steeper.

Natarajan and Iwasaki (1972) reported that when Eh/pH diagrams of sulphide mineral-water-oxygen systems are examined, a common line with a certain slope represents a passivated mineral surface due to the formation of oxides, or hydroxides and surface oxidation of sulphide minerals adversely affects their flotation behaviour. If the Eh-pH lines are taken as the reference line for the oxidation of the sulphide mineral surfaces, the approach of the measured Eh towards these lines will be an indication of the beginning of the formation of passive oxide or hydroxide layers at the surface, signalling a gradual decrease in their flotability (Natarajan and Iwasaki, 1972).

It has been noted by Ross and Van Deventer (1985) that the type of additive used to adjust the pH played significant role in some systems. But experimentally no significant differences were observed in the present study between H_2SO_4 and HCl as acid and NaOH and $Ca(OH)_2$ as alkaline regulator electrolytes on measured potential for each electrode.
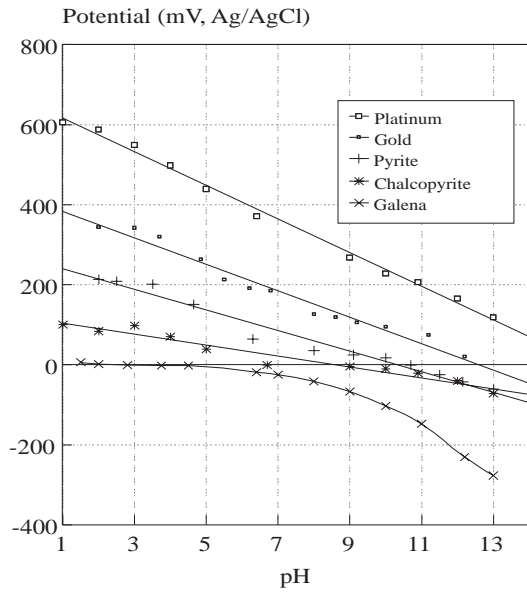
Figure 4. Electrodes potential as a function of pH.

Flotation of pure minerals

The flotation of sulphide minerals and their modulation has been a subject of investigation for many years. The influence of pH on the floatability of sulphide minerals is well known and it is usually used as one of the control parameters of flotation. However, detailed studies involving Eh/pH /flotation for pyrite, chalcopyrite, sphalerite and galena are relatively limited. The influence of pH on chalcopyrite, pyrite, a sphalerite and galena flotation with different xanthates as collectors and on pulp potential was experimentally observed in this part of the study. pH was varied from 2 to 13 with HCl and NaOH and pulp potential was recorded as a function of pH with a corresponding mineral electrode to the mineral being floated and also with a platinum electrode. A gold electrode was used in sphalerite flotation instead of sphalerite electrode because sphalerite was not conductive enough. Initial tests were carried out to determine the optimum amount of xanthates necessary to achieve maximum floatability. For chalcopyrite 0.7 mg/l, for pyrite and galena 2 mg/l and for sphalerite 1.5 mg/l were found to be the optimum collector amount. Recoveries of chalcopyrite, pyrite and sphalerite as a function of pH with sodium ethyl xanthate (NaEX), sodium isopropyl xanthate (SIPX), sodium isobutyl xanthate (SIBX) and potassium amyl xanthate (KAX) were also examined for the effect of xanthate carbon chain length.

Chalcopyrite

Chalcopyrite flotation was mainly independent of pH and xanthate type (Figure 5). Flotation of chalcopyrite from pH 2 to 13 is explained by electrochemical oxidation of xanthate to dixanthogen as well as by chemisorption of xanthate on chalcopyrite (Weiss 1985). Gardner and Woods (1979) proposed that in acid solution elemental sulphur would form and it renders the mineral hydrophobic. But not all the reported data are consistent. Ackerman *et al.* (1987) found that the recovery of chalcopyrite decreases continually as pH increases from 5 to 10.5.
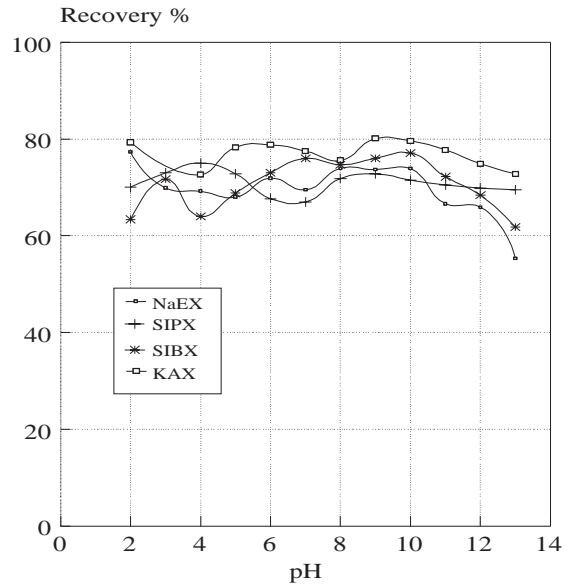

Figure 5. Recovery of chalcopyrite as a function of pH with different xanthates.

In present study potential varied from 200 mV to -150 mV with a chalcopyrite electrode and from 420 mV to -20 mV with a platinum electrode within a pH range of 2 to 13 (Figure 6). The presence of chalcopyrite particles and xanthate ions in pulp strongly affects the pulp potential where the solution potential was 100 to –30 mV for the chalcopyrite electrode and 600 to 180 mV for the platinum electrode for pH 2 to 13.

Pyrite

Control of the the solution pH is one of the most widely used methods for depressing pyrite flotation. Decreased flotability of pyrite in alkaline solutions was attributed to rapid decomposition of the collector form; preventing the formation of dixanthogen

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and the surface of pyrite would consist of a hydrophilic ferric hydroxide at high hydroxyl concentrations (pH > 11), (Weiss, 1985; Hoyack and Raghavan, 1987; Ackerman *et al.*, 1987a; Kocabağ *et al.*, 1990a; Fuerstenau *et al.*, 1968; Gardner and Woods, 1979). The electrochemical explanation of the depression of pyrite by an alkali in relation to the Eh/pH diagram reported by Janetski *et al.* (1977) is that at the lower pH values, pyrite oxidation occurs only at potentials much more anodic than the xanthate/dixanthogen couple reversible potential, 180 mV, and at these pH values oxygen reduction is rapid at the xanthate/dixanthogen potential. Hence a mixed potential system will operate in which xanthate will be oxidised much more easily than the pyrite surface. This will involve dixanthogen formation and a hydrophobic surface will occur. As the pH of the solution is increased, the surface is more readily oxidised. Under these conditions the mixed potential system becomes one of pyrite oxidation and oxygen reduction, xanthate is not oxidised, and the surface remains hydrophilic (Gardner and Woods, 1979; Janetski *et al.*, 1977). At pH 10.5 the rates of pyrite oxidation and oxygen reduction are found to be equal and opposite at a potential cathodic to that for xanthate oxidation. Hence only mineral will be oxidised at the mixed potential and flotation will be depressed (Janetski *et al.*,1977).
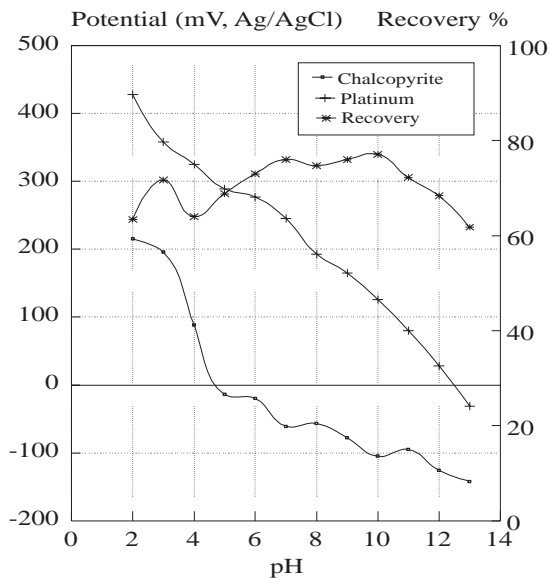

Figure 6. Recovery of chalcopyrite and pulp potential as a function of pH.

In Figure 7 the experimental results show that pyrite recovery increases gradually up to pH 5 and

becomes stable at pH 5 to 9 and then it drops dramatically as pH increases. At pH 5 pyrite is found to be very soluble and iron ions released from the surface of pyrite are found to have an important effect on collector adsorption where these ions can cause rapid oxidation of xanthate to dixanthogen (Ackerman *et al.*1987). At low pH elemental sulphur or dissolution of iron hydroxides from the mineral surfaces is found to be responsible for the increase in the recovery. Kocabağ *et al.*, (1990a) found the same pH range (at pH 5.5 to 9.5 maximum recovery was obtained) for pyrite flotation as the present study and stated that the decrease in recovery above pH 9 was due to the formation of $Fe(OH)_2$ on pyrite.

Pulp potential was also stable between pH 5 to 9 at about 60 mV where maximum recovery was obtained in this pH range. The difference between pyrite and platinum electrodes is 100 mV at low pH but the potential difference became closer at higher pH values. As shown in Figure 8, except ethyl xanthate, almost the same recovery curve was obtained with SIPX, SIBX and KAX as a function of pH. A similar but lower recovery trend occurred with ethyl xanthate.
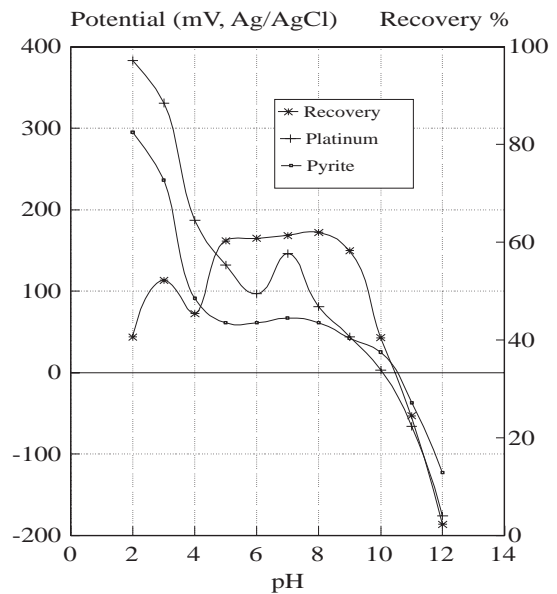

Figure 7. Recovery and pulp potential of pyrite as a function of pH.

Sphalerite

Some investigators have observed the flotation of sphalerite with xanthates in the absence of activators, while others have not. These differences may due to the sphalerite samples involved (Weiss, 1985).

The flotation response of sphalerite as a function of pH is shown in Figure 9. Recovery increased to about 67% up to pH 6 and thereafter dropped to less than 10% at pH 8. Bulut *et al.* (2000) found the best pH range for sphalerite to be below 6.5, but above this pH they also obtained a resonable flotation recovery. The iso-electric point of sphalerite was reported to be 6 in the presence of xanthates by Bulut *et al.*, (2000). The pulp potential varied as a function of pH with both platinum and gold electrodes from 100 mV to -200 mV and from 100 to -70 mV respectively. The potential difference between these two electrodes increased as pH increased, which is opposite to the behaviour of those electrodes in pure solution. When pulp potential is zero around pH 6.5-7, flotation drops dramatically. Marouf *et al.* (1986) found a recovery of sphalerite as a function of pH similar to that in the present study and also noted that, with amyl xanthate, sphalerite can be floated at pH values from 1 to 9, which is not observed in the present study.
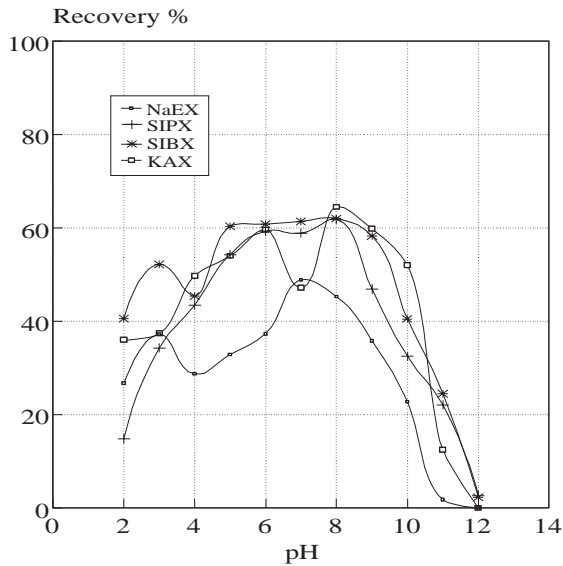

Figure 9. Recovery and pulp potential of sphalerite as a function of pH.

Figure 8. Recovery of pyrite as a function of pH with different xanthates.

As shown in Figure 10, as the carbon chain length of xanthate increases, recovery of sphalerite increases; using amyl xanthate increased the recovery of sphalerite but not the pH range of flotation. Fuerstenau *et al.* (1974) varied the pH in sphalerite flotation in the presence of various xanthates and observed no flotation with ethyl or iso-butyl xanthate. The present results are not in agreement with these observations.
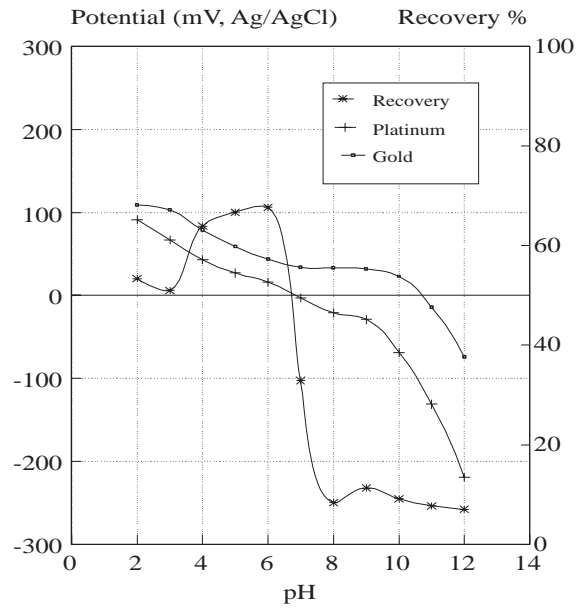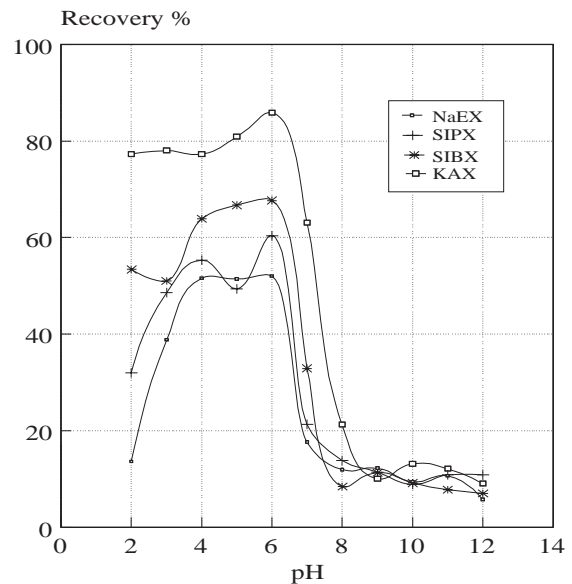

Figure 10. Recovery of sphalerite as a function of pH with different xanthates.

Galena

Figure 11 shows that galena flotation is possible below pH 7. Above this pH recovery dropped dramatically as pH increased. Potential changed only from 35 mV to –25 mV for pH 2 to 11 and dropped to –125 mV at pH 12. Again when pulp potential is zero at pH 7 flotation drops sharply. The measured

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potentials for both galena and platinum electrodes converged when pH increased. In the literature it has been noted that complete flotation of galena can occur from pH 2 to 10 (Weiss, 1985), whereas in the present study galena flotation was possible only below pH 7. Kocabağ *et al.* (1990b) obtained maximum recovery at about pH 5.5 and the minimum was obtained at neutral pH values with oxidised galena. Flotation of galena in acid solutions is explained by increased hydrophobicity with oxidation due to formations of sulphur on the surface by Kocabağ *et al.* (1990b), who observed increases in the contact angle with anodic oxidation at acid pH values 1-4. On the other hand, passing from acidic to neutral and alkaline solutions oxidation of galena takes place forming $Pb(OH)_2$ on the surface in addition to S^0, which decreases the hydrophobicity due to the formation of $Pb(OH)_2$ or metal-sulpoxy compounds (Kocabağ *et al.*, 1990b).

The difference in potential with platinum electrodes and galena electrodes as explained by Rand and Woods (1984) is that oxidation of xanthate on galena occurs at a more negative potential than on noble metals due to the formation of different reaction products: dixanthogen is produced on noble metals whereas chemisorbed xanthate and lead xanthate are additional products on galena. The difference in potential between the noble and mineral electrodes in pulp was at the beginning 75 mV and after getting the same potential, whereas in solution the difference was 600 mV at the beginning and 400 mV at the end. The presence of minerals results in a change in the composition of the solution due to the interaction of both xanthate and oxygen with galena particles. This shows that the platinum electrode starts to act as a slurry electrode and respond to the potential of the sulphide particles.

Summary and Conclusions

Potential is linearly dependent on the pH in solution. Only the galena electrode did not show this relationship, especially in an alkaline pH range. All electrodes gave very close potential in alkaline pH as explained in the literature due to passivation of the electrode surface with the formation of oxides and hydroxides. Because of the complexity of the flotation pulp, the Eh/pH responses of the electrodes were different in flotation pulps compared with simple solutions and mineral pulp where mineral electrodes show a smaller change than the platinum elec-

trode. This paper showed that mineral electrodes may respond to changes in the environment. Overall, if the pulp potentials are compared with flotation pulp and solution, it is obvious that they show a difference in slope. Table 2 shows the comparison of potential in solution, single mineral flotation and complex ore flotation for pH 9-12.
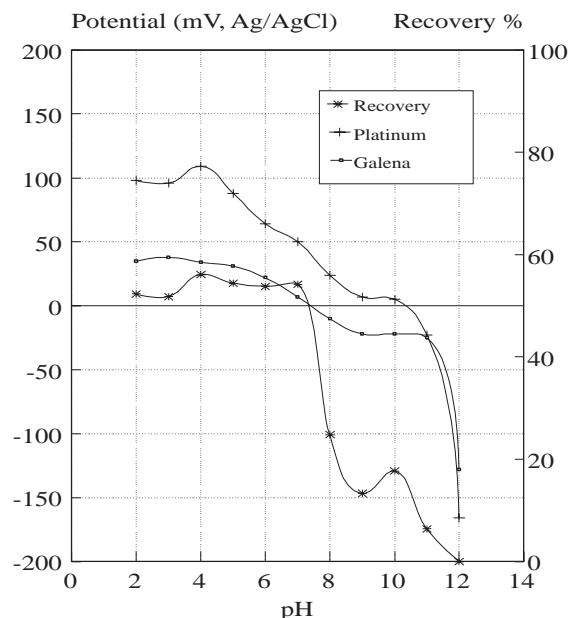

Figure 11. Recovery and pulp potential of galena as a function of pH.

In flotation systems, recoveries and potential show similar trends at high pH and pulp potential again depends on pH. Flotation of chalcopyrite is independent of pH and xanthate type. Also measured Eh values for both chalcopyrite and platinum electrodes were very different, unlike the others. In alkaline solution, pyrite, sphalerite and galena became poorly floatable because of decomposition of xanthate and formation of hydrophilic metal hydroxides on the sulphide mineral. The length of the C-chain had different effects on the flotation recoveries of chalcopyrite, pyrite and sphalerite, but mainly as it increases, recovery increases as well.

Pulp potential is an important electrochemical parameter that can be correlated with flotation results and it may determine the condition of the sulphide surface and prediction of regions of optimal flotation. The experimental work demonstrated the relevance of Eh-pH data for describing changes in the general chemical properties of pulp during a flotation process.

Table 2. Comparison of electrochemical potential (mV) in solution, pure chalcopyrite flotation and complex ore flotation with chalcopyrite (chp) and platinum (Pt) electrodes.

Eh/pH	pH 9		pH 10		pH 11		pH 12	
measurements	Pt	Chp	Pt	Chp	Pt	Chp	Pt	Chp
Solution	270	-10	220	-20	200	-30	180	-50
Pure mineral float.	170	-80	125	-100	80	-100	25	-125
Complex ore float.*	80	-25	50	-50	10	-70	-100	-115

*From reference Göktepe and Williams, 1995.

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