



## EFFECT OF ELECTROCHEMICAL CONTROL ON SELECTIVE FLOTATION OF COPPER AND ZINC FROM COMPLEX ORES

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### ABSTRACT

*Effects of grinding media and electrochemical conditions on selective flotation of copper and zinc were investigated using three different complex sulphide ore samples. Grinding medium had a strong effect on the electrode potentials and the flotation results of the three ore types studied. Copper recovery increased by 80% within a narrow potential range in the flotation of the Pyhäsalmi ore that represented an "easy" ore type whereas only 15% increase in copper recovery occurred in the flotation of the "difficult" Aljustrel ore. The "moderate" Grong ore responded to the potential as with the Pyhäsalmi ore but the potential for complete depression of copper was 100 mV lower. The optimum potential range of the chalcopyrite electrode was +50 to +150 mV (vs. SHE) in the copper flotation of the complex sulphide ore types studied. The optimum potential of the sphalerite composite electrode in the zinc flotation of the three ore types was that obtained using full aeration. The Aljustrel and the Grong ores resulted in the highest Zn grades, using low potential (-150 mV) at the activation stage, whereas the Pyhäsalmi ore yielded the best recovery and grade at "air set" potentials. © 1997 Elsevier Science Ltd. All rights reserved.*

### Keywords

Flotation collectors; flotation activators; froth flotation; grinding; redox reactions

### INTRODUCTION

It is now evident that electrochemistry plays an important role in controlling the interaction between collectors and sulphide mineral surfaces. A number of fundamental studies have been carried out to show that sulphide minerals can be recovered within certain ranges of electrode potential typical of the specific mineral [1,2]. However, application of electrochemical control in the mineral processing industry has not been very widespread due to the practical problems involved in process conditions. There has also been difficulty in converting the fundamental knowledge of electrochemical processes at mineral surfaces to useful parameters in mineral processing plants. The correlation between single mineral electrodes studies under well-defined laboratory conditions and the real  $E_h$  behaviour under plant conditions is not always

straightforward. Consequently, mineral processors need sufficient education in electrochemistry for successful electrochemical control of mineral processes. The practical problems in electrochemical control include long-term stability and fouling of electrodes as well as the difficulty in adjusting the potential at the required level.

Monitoring of potential with noble metal electrodes [3], such as platinum, is commonly carried out in laboratory flotation testwork and sometimes also at full-scale processes. A more sophisticated way of potential measurement can be carried out with sulphide mineral electrodes prepared from pure minerals or minerals from the particular orebody [4,5]. Air is the most common gas used in flotation and the minerals in the flotation pulp are subjected to free aeration. Most of the sulphide flotation plants operate at 'air set' potentials that are usually +100 to +300 mV vs. SHE. To operate in less oxidising conditions, different reducing agents can be used for  $E_h$  control, such as sodium sulphide, hydrazine, sodium dithionite and sulphur dioxide. Application of reducing agents in aerated slurries usually leads to high reagent consumption due to the fact that they are excessively consumed by the oxygen in the flotation gas.

It is now well established that grinding influences the chemistry of the subsequent flotation stages [6–9]. Iron present in the grinding medium plays an important role in providing galvanic contacts between mineral grains and changing the redox conditions through iron–iron(II)–iron(III) couples. Nevertheless, the effect of grinding medium on the flotation of complex ores has not been studied very extensively although the redox conditions in autogenous grinding are known to be different from those in normal steel grinding.

Selective flotation of complex sulphide ores poses several difficulties due to mineralogical characteristics, incomplete liberation and flotation chemistry differences between the ores. Complex mineralogy, connected with difficult flotation chemistry, has hindered the economic beneficiation of several large ore bodies throughout the world. The present study is focused on the selective flotation of copper and zinc from three different pyrite-bearing complex ores: Pyhäsalmi (Finland), Grong (Norway) and Aljustrel (Portugal). The major objective of this work was to study and compare the effect of electrochemical control on the recovery and grade of copper and zinc using mineral electrodes as electrochemical sensors in the slurry.

## EXPERIMENTAL

### Materials

The Aljustrel ore sample was provided by Pirites Alentejanas S/A, Aljustrel, Portugal. The Grong ore sample was provided by the Grong mine, Norway and the Pyhäsalmi ore sample by the Pyhäsalmi mine of Outokumpu Mining Oy, Finland. The lump samples were jaw crushed and dry screened to a grain size of 100% –1 mm and stored in a freezer under nitrogen. For flotation tests one kilogram of the crushed ore was ground in a normal steel mill or in a stainless steel mill.

### Flotation experiments

Grinding for the flotation tests was carried out on the –1 mm crushed ore samples either in a stainless steel mill or a normal steel mill. The flotation experiments were performed with 1 kg samples in a 4 litre cell using the control system described in this paper. Alkyl xanthates were used in the copper and zinc flotation and copper sulphate for activation in the zinc flotation. The copper and zinc flotations were carried out at pH 11.5 and 12, respectively. Only rougher flotation tests were performed analysing the rougher concentrates at 1, 3, 6 and 10 min from starting the gas flow. Cumulative recoveries and grades were calculated from the masses and chemical analyses of the concentrates and the tailing. The products from the flotation experiments were analysed for Cu, Zn, Pb, Fe, As and S.

In the copper flotation experiments, potential was monitored using gold, platinum, glassy carbon, pyrite, pyrrhotite, chalcopyrite, galena and arsenopyrite electrodes. In the zinc flotation stage, the potential of a sphalerite-composite electrode was also monitored. The mineral electrodes were prepared by mounting a rod-shaped 10 x 6 mm mineral sample into a plastic tube using epoxy after attaching a copper wire to one face of the sample using conducting epoxy. The diameter of the electrode face was then approximately 6

mm. The sphalerite composite electrodes were prepared by Outokumpu Research Oy, Pori, Finland using two different sphalerites: sphalerite A containing 0.26% iron and B containing 8.54% iron. The response of the sphalerite electrode of the lower iron content proved to be more useful in controlling the zinc flotation. A calomel reference electrode was used but all potentials reported are vs. the standard hydrogen electrode assuming that the standard electrode potential of the calomel electrode is 0.244 V. The electrode surfaces were renewed prior to each flotation test using 600 grade emery paper. The electrodes were placed into the slurry, mounted in a special sample holder attached to the flotation cell and were immersed at a depth of about 50 mm below the froth–slurry interphase in the cell.

### Method of potential control

The 'advanced flotation system' developed by Mineral Processing of the Technical Research Centre of Finland was used in the flotation tests. The system has been described in detail previously [10]. In the present work, nitrogen was used as the major flotation gas and air as the oxidative gas. After grinding in a normal steel mill, the potential level was too low for significant flotation recovery of sulphide minerals or precious metals. In this flotation system, air was first added in the flotation gas so that the desired potential level was reached. When the desired potential level for the flotation of an individual mineral was obtained, the aeration was stopped and an inert flotation gas, usually nitrogen, was used. After this stage, air was fed to the flotation gas stream through a titrator unit to maintain the desired potential level. With this control system, potential could be adjusted within 2 to 5 mV during the flotation period.

## RESULTS AND DISCUSSION

### Composition of the ore samples

The chemical compositions of the Aljustrel, Grong and Pyhäsalmi ore samples are presented in Table 1. The calculated mineralogical compositions are shown in Table 2. All the complex sulphide ores studied contained over 50% pyrite, the highest content being in the Aljustrel ore, 84.7%, and the lowest in the Grong ore. The Aljustrel ore represented the most complex ore type, containing several copper minerals such as chalcopyrite, bournonite and tetrahedrite, as well as two lead minerals galena and bournonite. In addition, the Aljustrel ore contained arsenopyrite. The mineralogical composition of the Grong ore was less complex than that of the Aljustrel ore, the major sulphides being chalcopyrite, sphalerite, pyrrhotite and pyrite. The Pyhäsalmi ore had the simplest sulphide mineralogy containing three major sulphides chalcopyrite, sphalerite and pyrite.

TABLE 1 Chemical composition of the ore samples.

Ore (w%)	Aljustrel	Grong	Pyhäsalmi
Cu	0.91	1.80	0.85
Zn	2.60	0.91	2.25
Pb	0.88	0.02	0.09
Fe	44.6	33.3	36.2
S (XRF)	51.4	29.8	42.2
S (LECO)	48.0	31.6	40.3
As	0.55	0.34	0.02
SiO <sub>2</sub>	2.4	18.2	13.0

**TABLE 2** Calculated mineralogical composition of the ore samples.

MINERAL (w%)	ALJUSTREL	GRONG	PYHÄSALMI
Chalcopyrite	2.56	5.20	2.46
Sphalerite	4.25	1.52	3.86
Galena	0.91	0.02	0.11
Pyrite	84.7	52.7	74.0
Pyrrhotite	<0.1	2.95	<0.1
Arsenopyrite	1,19	<0,1	<0,1
Tetrahedrite	0,04	N/A	N/A
Bournonite	0,23	N/A	N/A
Barite	N/A	N/A	3,35
Others	6,15	37,6	16,2

Due to the dissemination of the sulphide mineral grains the grinding fineness required to obtain satisfactory concentration varied considerably among the three ore types (Table 3). The optimum grinding time for the Aljustrel ore was approximately 45 minutes whereas the corresponding grinding times for the Grong and the Pyhäsalmi ores were 18 and 20 minutes, respectively. The d80 value for the Aljustrel ore was 28  $\mu\text{m}$ , for the Grong ore 67  $\mu\text{m}$ , and for the Pyhäsalmi ore 110  $\mu\text{m}$ .

**TABLE 3** Grinding fineness of the ore samples in the flotation studies.

Particle size passing (%)	Aljustrel (45 min)	Grong (18 min)	Pyhäsalmi (20 min)
125	99.9	97.2	87.5
90	99.7	92.7	69.7
75	99.0	86.1	59.5
63	98.0	77.5	59.5
45	92.8	62.1	37.5
32	84.1	48.7	29.6
20	68.4	34.7	23.6

### Effect of grinding media on the copper flotation

Grinding media had a dramatic effect on the measured electrode potentials in the slurry. According to potentials measured with a chalcopyrite electrode for all the ore types studied, grinding in a normal steel mill generated conditions that were much more reducing than for grinding in a stainless steel mill (Figure 1). Due to aeration in the flotation stages, the potentials shifted in a more oxidising direction (stages 8–11) and after 15 minutes flotation the potential difference was usually only 20 to 30 mV.

Depending on the ore type, the potential difference between the two grinding media was 100 to 250 mV after grinding (stages 1–2) being up to 400 mV after conditioning with lime at pH 11.5 (stage 4). The greatest differences between the normal steel and the stainless steel mills were observed on the Aljustrel

ore. The Grong ore showed slightly smaller potential differences compared to the Aljustrel ore, while the potential differences were clearly smallest on the Pyhäsalmi ore, being about 100 mV at stage 1 and 150 mV at stage 4. (Figure 2). Interestingly, when lime was added directly in the grinding stage to achieve alkaline conditions for the copper flotation, there was very little difference between the two grinding media. Unlike the normal steel grinding, there is only a limited potential range in the stainless steel grinding for electrochemical control of flotation using nitrogen and air.

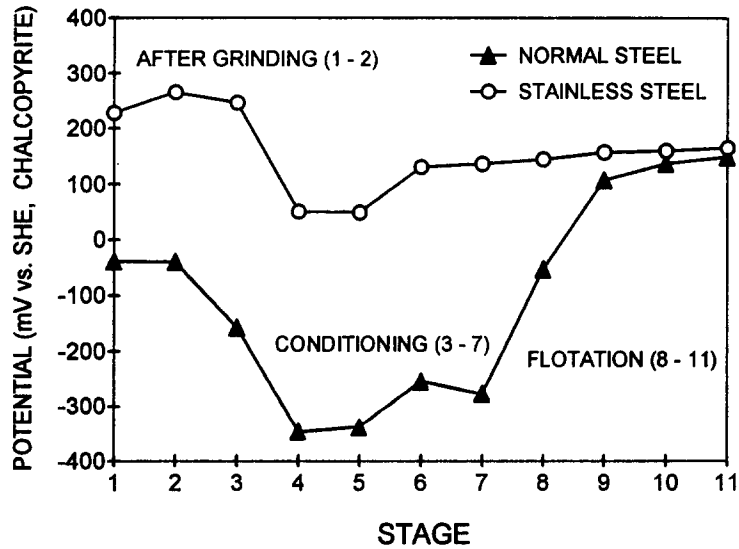


Fig.1 Potential of chalcopyrite electrode at various stages of the flotation test of the Aljustrel ore after grinding in a stainless steel and in a normal steel mill. Sulphur dioxide, lime and sodium isobutyl xanthate were used in the conditioning stage. Flotation time 1+2+3+4 min.

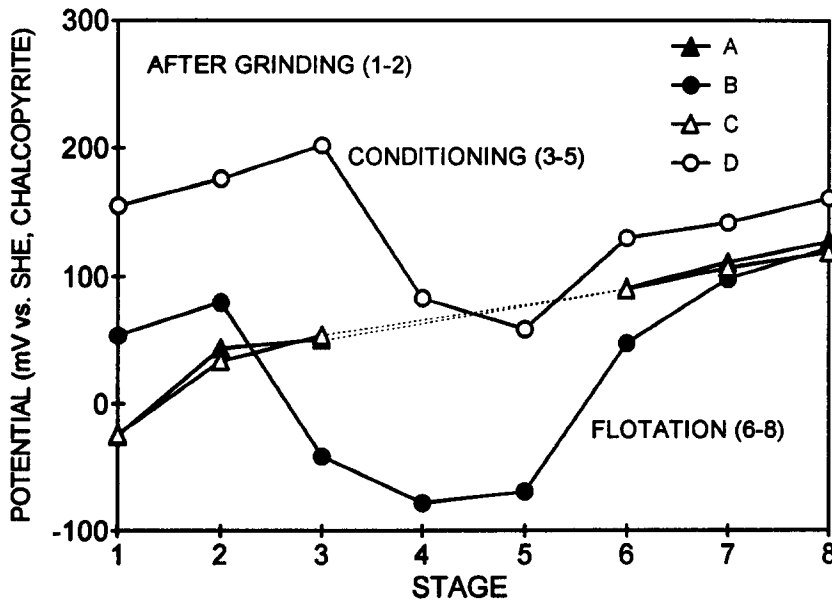


Fig.2 Potential of chalcopyrite electrode (vs. SHE) at various stages of the flotation test of the Pyhäsalmi ore. Normal steel mill: lime and zinc sulphate in grinding (A); Normal steel mill: lime and SO<sub>2</sub> in conditioning (B); Stainless steel mill: lime and zinc sulphate in grinding (C); Stainless steel mill: lime and SO<sub>2</sub> in conditioning (D). Sodium isobutyl xanthate was used as the collector.

The flotation behaviour of copper and zinc minerals in the complex ores was greatly affected by the grinding media. Using the normal steel medium, copper recovery was low at low potentials but increased when the potential became more oxidising (Figure 3). It was observed that copper grade was low at the low potentials after grinding in the normal steel mill. This was due to an excessive recovery of pyrite diluting the copper concentrate. As cumulative recoveries and grades were examined the copper grades remained low even when recoveries were high after the potential was elevated to more anodic (positive) values. Similar phenomena, where pyrite pre-float was obtained at low potentials, were observed for the three ore types. The tendency of pyrite to float at low potentials could be due to "incipient" oxidation of pyrite that occurs at much lower potentials than would be expected by bulk thermodynamics [11]. When lime was added into the grinding stage of the Pyhäsalmi ore, copper grades and recoveries were almost equal in the normal steel and stainless steel grinding (Figure 4).

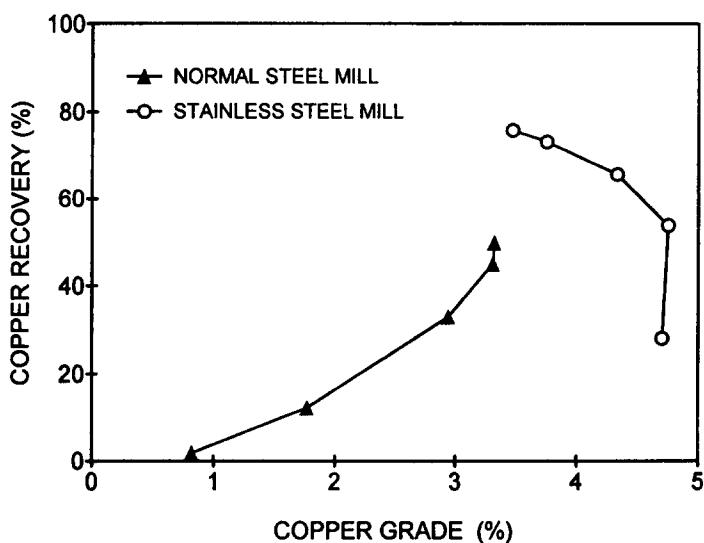


Fig.3 Relationship between cumulative copper grade and recovery in the copper flotation of the Aljustrel ore after grinding in a stainless steel and in a normal steel mill. Sulphur dioxide, lime and sodium isobutyl xanthate were used in the conditioning stage. Flotation time 1+2+3+4 min.

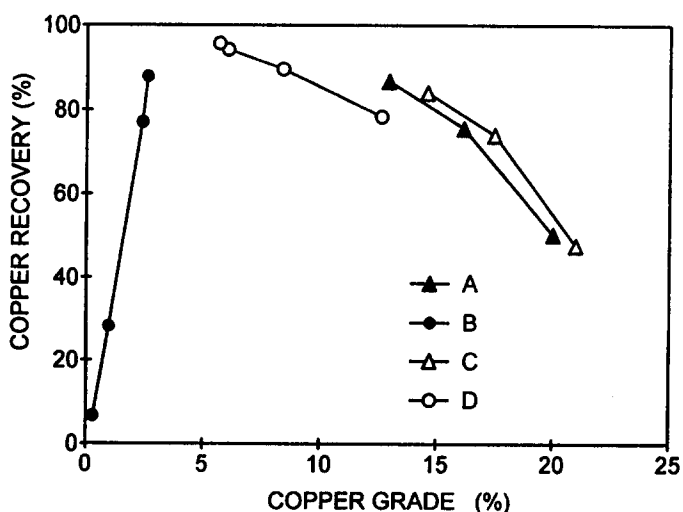


Fig.4 Relationship between cumulative copper grade and recovery in the copper flotation of the Pyhäsalmi ore. Normal steel mill: lime and zinc sulphate in grinding (A); Normal steel mill: lime and SO<sub>2</sub> in conditioning (B); Stainless steel mill: lime and zinc sulphate in grinding (C); Stainless steel mill: lime and SO<sub>2</sub> in conditioning (D). Sodium isobutyl xanthate was used as the collector.

In the more favourable electrochemical conditions generated in the stainless steel medium, copper recovery was high from the first minute of the flotation stage. However, due to very oxidising conditions, the selectivity of flotation can be poorer after grinding in a stainless steel medium than after grinding in a normal steel medium [10].

Metallic iron, and its equilibria with iron(II) and iron(III) ions, plays an important role in controlling the electrochemical conditions in the flotation of complex sulphide ores. These effects are more pronounced when the grinding fineness is high (Aljustrel ore) but also play an important role with ores of coarser grinding (Pyhäsalmi). When lime is used in the grinding stage the effect of iron is largely eliminated probably due to the solution equilibrium favouring the formation of iron hydroxides. In separate studies [12] it was observed that the addition of iron powder and ferrous sulphate created results comparable to grinding in a normal steel mill.

### Effect of electrochemical control on the copper flotation

When the conditioning and flotation stages (stages 7 to 11) were carried out using a special potential control system, the oxidation potential could be maintained at the desired level and the effect of potential on copper flotation could be studied in more detail. This was particularly interesting when grinding was carried out using a normal steel mill with a large available operation range. As stated earlier, there is only a narrow potential range available after stainless steel grinding.

Copper recoveries in the flotation of the three ore types are shown as a function of potential in Figure 5, showing that the effect of potential was very sharp on copper flotation from the Pyhäsalmi ore, whereas the response of the Aljustrel ore to potential control was sluggish. One reason for the different behaviour was that copper recovery from the Aljustrel ore was relatively high even at very low potentials, where only low copper recoveries were obtained from the Grong and the Pyhäsalmi ores. For all three ores, copper grade was markedly increased at higher potentials (Figure 6).

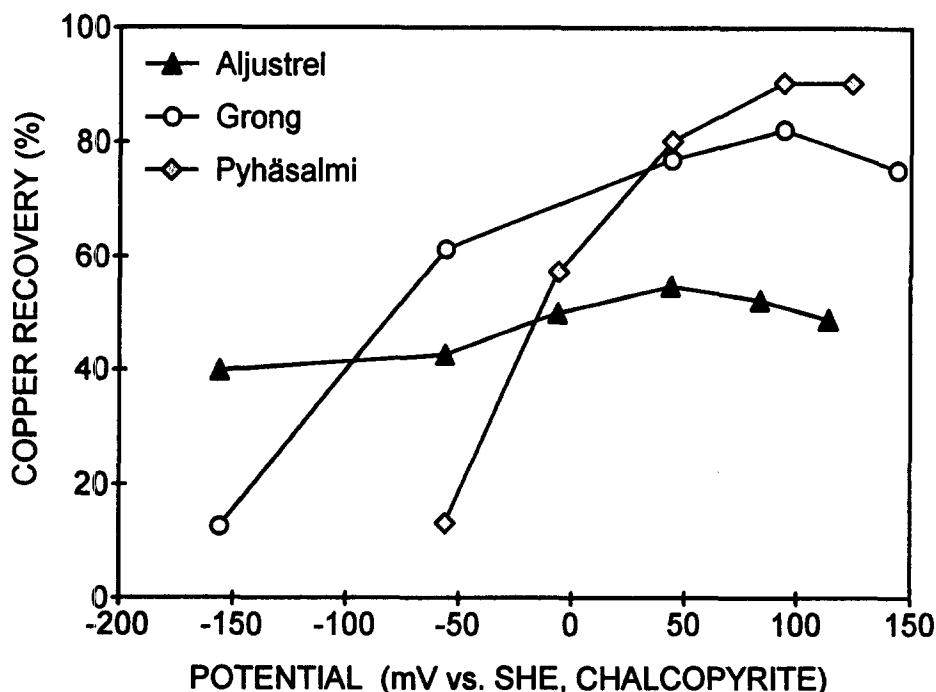


Fig.5 Relationship between cumulative copper recovery and potential after 6 min copper flotation of the three ore samples when normal steel mill was used in grinding and xanthate as the collector at pH 11.5.

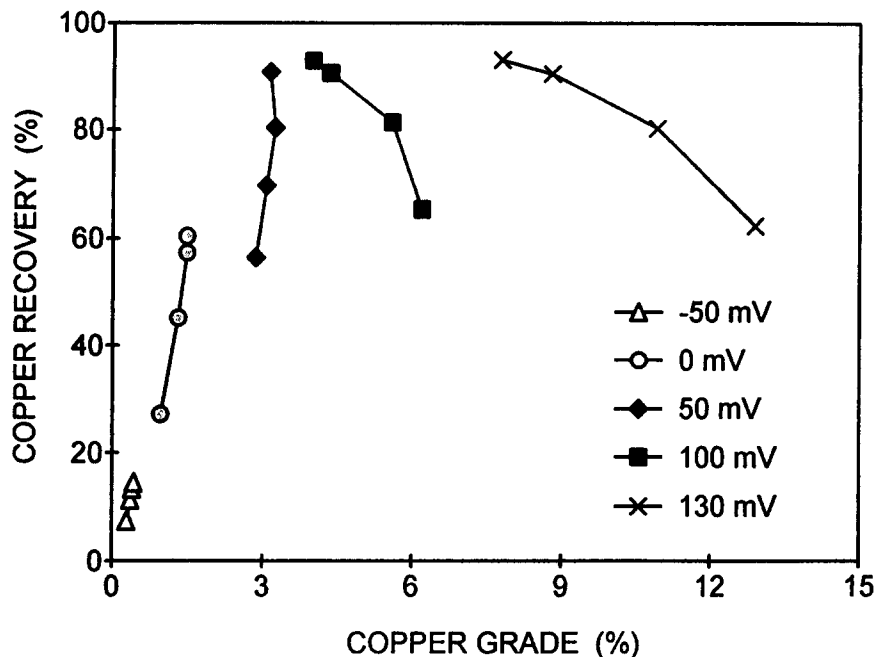


Fig.6 Relationship between cumulative copper grade and recovery in the copper flotation of the Pyhäsalmi ore after grinding in a normal steel mill using isobutyl xanthate. Flotation time 1+2+3+4 min.

Zinc recovery increased in the copper flotation as a function of potential up to +50 mV (Figure 7) but decreased at potentials of +100 mV or higher. This indicates that the maximum zinc recovery is obtained

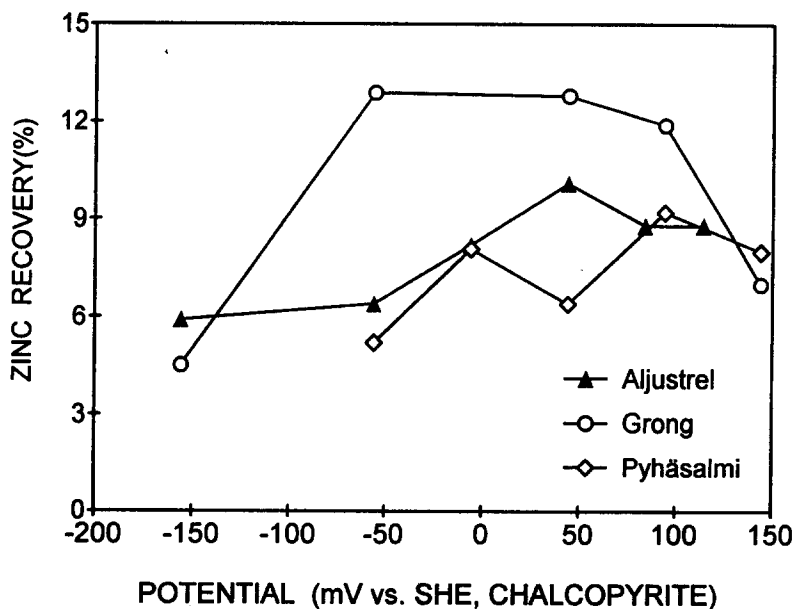


Fig.7 Relationship between cumulative zinc recovery and potential after 6 min copper flotation of the three ore samples. Normal steel mill was used in grinding and xanthate as the collector at pH 11.5.

in the same potential range as observed for copper. Copper recovery decreased at very high potentials but zinc recovery decreased even more, providing better selectivity for copper flotation at more anodic



potentials. Sulphur recoveries (Figure 8) attained maximum values at  $-50$  to  $0$  mV but decreased steadily at more oxidising conditions as a function of potential. Due to the high pyrite content of these ore samples the behaviour of sulphur is parallel to the behaviour of pyrite indicating that pyrite will be depressed in oxidising conditions.

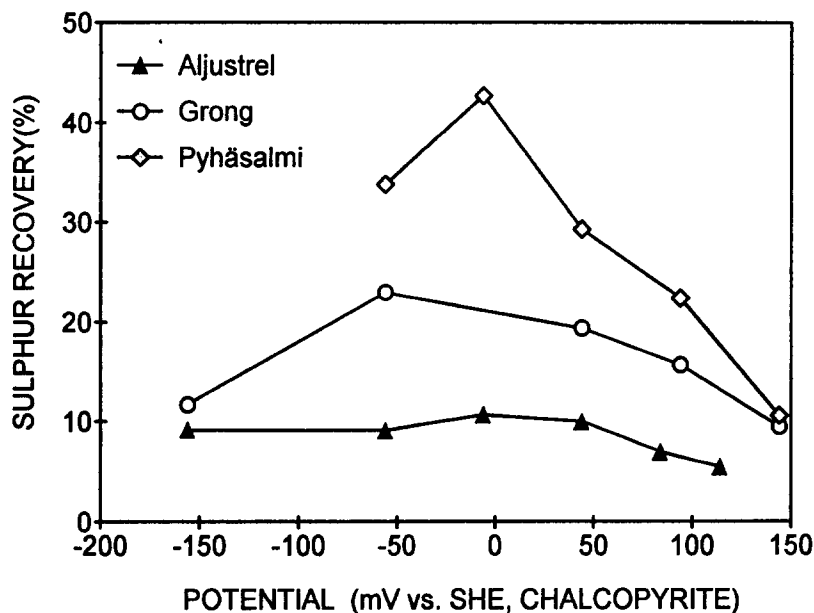


Fig.8 Relationship between cumulative sulphur recovery and potential after 6 min copper flotation of the three ore samples. Normal steel mill was used in grinding and xanthate as the collector at pH 11.5.

In general, the optimum potential for copper flotation yielding the highest selectivity for the ore types studied was in fully aerated systems,  $+100$  to  $+150$  mV (SHE), measured with a chalcopyrite electrode. Simultaneous potential measurements using a platinum electrode give  $+70$  to  $+90$  mV higher potentials than the chalcopyrite electrode, i.e.  $+170$  to  $+240$  mV. An important finding for all the ore types studied was that the recovery of pyrite decreased as a function of electrode potential, starting at  $-100$  to  $0$  mV, being lowest in fully aerated solutions at  $+100$  to  $+150$  mV.

The potential of maximum copper recovery is close to the potential of dixanthogen formation of isobutyl xanthate which is  $+117$  mV at a xanthate residual concentration of  $10$  mg/l [13]. The onset of flotation, however, occurs at potentials that are considerably lower than the formation potential of dixanthogen. This is likely to be related to chemisorption reactions of xanthate forming a copper surface compound. Chalcopyrite has also shown to exhibit strong self-induced flotation which complicates the effects of xanthate. It has been shown by several authors that self-induced flotation starts at about  $0$  mV and maximum recovery is attained at about  $+200$  mV. A characteristic current peak was observed on voltammograms for chalcopyrite in the absence of xanthate at about  $0$  mV [14]. Unfortunately, the role of self-induced flotation cannot be solved based on this work because xanthate was used throughout the study.

The relationship between electrode potential and flotation recovery of this work was compared with the work of Richardson and Walker who presented results of single mineral studies of chalcopyrite under electrochemically controlled conditions using ethyl xanthate [2]. In their study, the lower flotation edge was about  $+50$  mV and maximum recovery occurred at  $+250$  mV. For xanthates of longer hydrocarbon chain used in this work, the potential is expected to shift to more negative values by  $70$ – $100$  mV [13]. Consequently, the potentials of Richardson and Walker come very close to those measured in this work. This indicates that the flotation response of potentiostatically controlled single minerals can be in good agreement with the flotation response of chalcopyrite from complex ores monitored by a chalcopyrite electrode in the mineral slurry.

Pyrite displayed floatability particularly between  $-50$  mV and  $0$  mV, this potential range being clearly below the formation potential of dixanthogen. On the other hand, previous electrochemical studies indicate that the hydrophobisation of pyrite is weak due to xanthate oxidation to dixanthogen at high pH [16]. The floatability of pyrite can arise from the incipient oxidation of the mineral surface generating hydrophobic sulphur species at potentials below the formation potential of dixanthogen [11]. There are indications that iron xanthates can also increase the hydrophobicity of pyrite below the formation potential of dixanthogen. Despite the presence of possible hydrophobic species, hydrophilic iron hydroxides and sulfur–oxygen species seem to reduce the hydrophobicity of pyrite leading to its depression at potentials above  $-100$  mV. Recently, iron(III) hydroxides were observed on pyrite after treatment at oxidising potentials using XPS measurements [17].

### Effect of electrochemical conditions on the zinc flotation

The effect of electrode potential on the flotation of zinc minerals has seldom been studied, mainly due to problems encountered in the potential measurement with poorly conducting mineral electrodes [15]. A specially designed composite electrode, where sphalerite was embedded in a conducting matrix, provided interesting information on the zinc flotation stage of the complex sulphide ores. It also became evident that neither metal electrodes nor other mineral electrodes can provide similar information to the sphalerite composite electrode (Figure 9). Unsuccessful attempts were made to use a platinum electrode, assuming that its potential differs from the potential of the sphalerite composite electrode by a constant value.

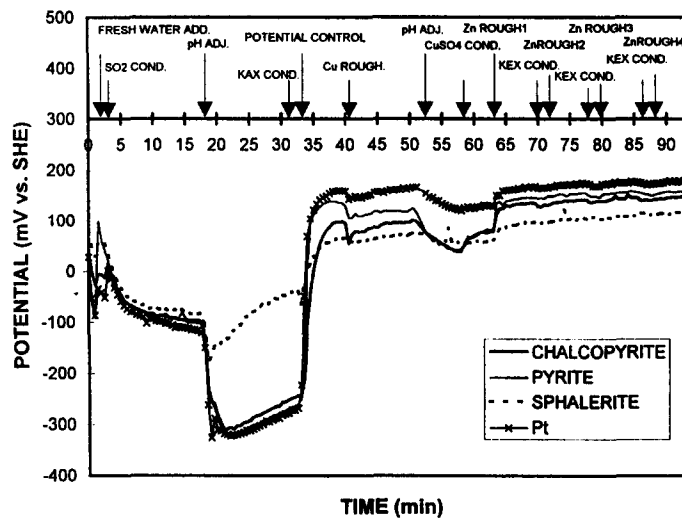


Fig.9 Potentials of four electrodes in the laboratory flotation of the Aljustrel ore.

When the flotation stage was carried out at "air set" potentials,  $+150$  mV, measured with a sphalerite composite electrode, zinc recovery and zinc grade were generally high for the three ore types (Figure 10). The zinc flotation results indicated that, in addition to the potential control in the flotation stage, the potential in the activation stage had a separate effect on the zinc flotation. For the ore types studied, zinc recovery increased as a function of the activation potential. The Pyhäsalmi ore showed no marked change in zinc recovery above  $-50$  mV, but a slight decrease in copper and sulphur recoveries was observed, making the highest potential ( $+150$  mV) the most favourable for the zinc flotation. Zinc recovery from the Grong ore increased steeply as a function of electrode potential and the highest recoveries were obtained at the "air set" potential, but the highest Zn grades were obtained at  $-150$  mV which was the most reducing activation potential studied. Although Zn recovery increased slightly as a function of the activation potential, superior Zn grades were also obtained for the Aljustrel ore at the lowest activation potential,  $-150$  mV (Figure 11).

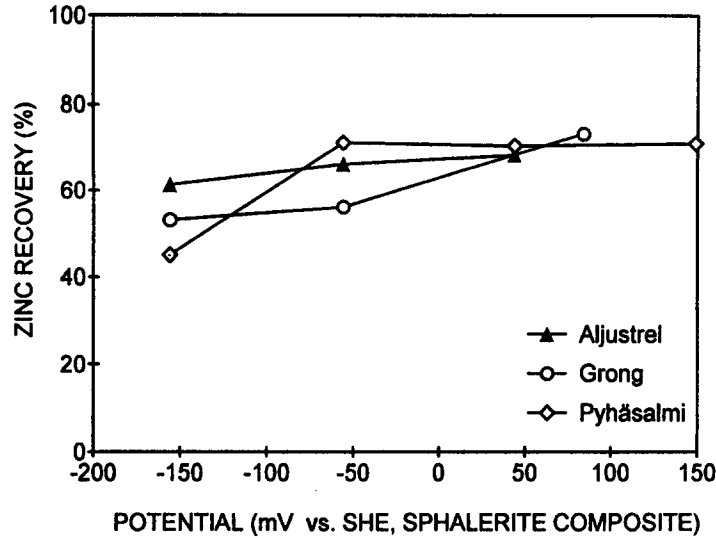


Fig.10 Effect of potential of the activation stage on cumulative zinc recovery after 6 min zinc flotation of the three ores. Flotation was carried out at "air set" potential at pH 11.5 to 12 using xanthate as the collector and copper sulphate as the activator.

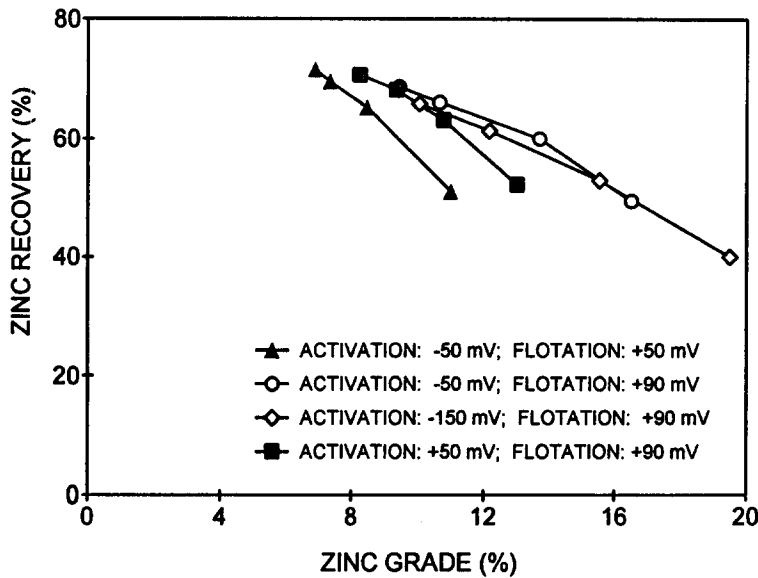


Fig.11 Relationship between cumulative zinc grade and recovery in the zinc flotation of the Aljustrel ore. Flotation was carried out at pH 11.5 to 12 using xanthate as the collector and copper sulphate as the activator. Flotation time 1+2+3+4 min.

Electrochemical conditions in the activation and conditioning stages have separate roles in the zinc flotation of the complex ores. There are findings in the literature that like the chemisorption of collectors activation is also an electrochemically controlled process [14]. This means that the mechanism of activation does not solely comprise ion exchange, where copper ions replace zinc ions from the surface of sphalerite, but copper(II) can be reduced to copper(I) at the surface. The electrochemistry of the activation process of sphalerite requires further research efforts.

## CONCLUSIONS

Laboratory-scale flotation studies were carried out to investigate the effect of grinding media and mineral electrode potentials on the flotation of copper and zinc from three different complex sulphide ore types.

- The type of grinding medium had a strong effect on the electrode potentials and the flotation results of all three ore types studied. Lime added into the mill largely eliminated the effects of grinding media.
- Copper recovery increased by 80% within a narrow potential range in the flotation of the Pyhäsalmi ore, which represented an "easy" ore type whereas only 15% increase in copper recovery occurred in the flotation of the "difficult" Aljustrel ore. The "moderate" Grong ore responded to the potential like the Pyhäsalmi ore but the potential for complete depression of copper was 100 mV lower.
- The optimum potential range of the chalcopyrite electrode was +50 to +150 mV (vs. SHE) in the copper flotation of all the complex sulphide ore types studied.
- The optimum potential of the sphalerite composite electrode in the zinc flotation of the three ore types was that obtained using full aeration.
- The Aljustrel and the Grong ores resulted in the highest Zn grades using low potential (–150 mV) at the activation stage whereas the Pyhäsalmi ore yielded the best recovery and grade at "air set" potentials.
- The chalcopyrite electrode displayed an excellent response to metallurgical performance in the copper flotation of complex ores and the sphalerite composite electrode had good response in the zinc flotation.
- Electrochemical control using mineral electrodes and flotation gases provides an excellent tool for laboratory-scale flotation studies and wider use of electrochemical control can be expected in industrial separation processes.

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