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# Electrochemical recovery of copper in the presence of contaminant ferrous ions

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The aim of this study was to collect specific electrochemical conditions for separate copper deposition from mixed copper-iron model electrolytes. The investigation is devoted for further utilization of copper rich industrial wastes. Reasonable, the composition of studied model electrolytes was considered with real solid and leach secondary wastes. The electroextraction of copper has been studied in the presence of ferrous ions from acidic sulphate electrolytes by means of potentiodynamic and galvanostatic methods. The potentiodynamic behaviour of copper was tested by adding or inclusion the ferrous ions from the investigated system. The obtained experimental results show that the harmful effect of ferrous ions on copper deposition could be minimized or modulated favorable without removing the iron contamination only by increaseng the cupric content. Ferrous ions could be separated potentiodynamically from copper ions by restriction of the range of the scanning potential. In all cases the presence of ferrous ions at the potentiodynamic studies reduces the anodic peaks of copper dissolution. In galvanostatic regime the monitored current efficiency declines abruptly with increase of ferrous ions concentration. At sufficiency high cupric ion concentrations this important parameter was higher than 90% and practically was independent from the presence of ferrous ions. The copper coatings obtained from equally mixed $Cu^{2+}/Fe^{2+}$ electrolytes were dark red in color, brittle and powdery. In comparison at higher $Cu^{2+}/Fe^{2+}$ ratio 5:1 the coatings were light red in color, smooth and semi-bright in appearance. In both cupric ions concentrations the coatings were composed only by copper metal phase proven by powder diffraction and scanning electron microscopy methods.

***Keywords*****:** selective electroextraction; cupric ions; ferrous ions, cyclic voltamperometry; galvanostatic deposition, SEM, XRD

## INTRODUCTION

After a number of pyro or hydrometallurgical ore treatments, large amounts of wastes with high metal content remain. The waste product known as “blue powder” that results from condensing furnace gases during the thermometallurgical processing of non-ferrous ores contains large amounts of zinc, iron and copper compounds [1, 2]. Lead-zinc cake, produced as a result of zinc concentrate leaching, contains extractable zinc, lead, copper and iron compound residues [3]. Similar composition could be attributed to other specific wastes as “Flue dusts” at a secondary copper smelter treated in the electrowinning zinc plant [4]. The industrial “Zinc residue”, which is a mixture of zinc scrap and zinc dross resulting from the zinc cathode industry contains large amounts of valuable zinc in the presence of copper and iron contaminants [5]. The residue discarded as a “cake” from a Waelz kiln processing zinc-lead carbonate ores contains mainly lead, zinc and iron compounds [6]. There are numerous papers concerning the recovery of metals contained in low concentrations in the wastewater produced in the metal plating industry by means of liquid extraction or other chemical methods. These methods, however, are inapplicable to the extraction of metals from solid wastes like powders, cakes, dusts, drosses, scraps, etc., where the content of non-ferrous metals is much higher.

We found in the literature only three papers that discuss in a systematic way the influence of ferric and ferrous ions on the electroextraction of copper from electrolytes with low metal concentration. By means determination of polarisation curves Dew et al. studied the effect of ferrous and ferric ions on the cathodic and anodic reactions for dilute acid copper sulphate electrolytes with varying concentrations and mixtures of reacting species [7]. The results describe the limiting rate of mass transfer for the deposition of copper and reduction of ferric ions. The cathodic reduction of ferric to ferrous ions results in a mixed potential at the cathode during copper electrowinning, and consequently the current efficiency of deposition would decrease significantly at high ferric concentration. Re-oxidation of ferrous to ferric ions at the anode would increase the effect of the ferric ions concentration on the current efficiency [7]. In a following study the same authors also investigated the effect of ferrous and ferric ions on the efficiency

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of copper recovery from solutions containing less than 2 g/L cupric ions, with an equivalent or greater concentration of iron as ferrous and ferric ions [8]. Experiments showed that the current efficiency decreased in proportion to the increase in the ferric ions concentration. The work has shown that by using of the Chemelec cell can achieve reasonable efficiencies for direct electrowinning from dilute leach liquors [8]. Das et al. studied the influence of ferric ions concentration (varied from 0.5 to 6.0 g/L) on the current efficiency, power consumption and cathode quality during copper electrowinning in an open channel cell [9]. A decrease in current efficiency started with increasing ferric ions concentration at each of the flow rates studied. When ferrous ions were added to $Cu^{2+}$-$Fe^{3+}$ electrolyte, the harmful effect of ferric ions on the current efficiency was reduced. More than 90% current efficiency may be achieved if the $Fe^{3+}$/ $Fe^{2+}$ ratio is maintained at less than or equal to 1. A marginal increase in the current efficiency was observed during increase in $Cu^{2+}$ concentration. The authors referred this tendency to increased solution viscosity at higher cupric ion concentrations and thus retarding the distribution of ferric ions over the cathode surface [9]. A decrease in current efficiency was observed when the ferric ions concentration were increased from 1.0 to 2.0 g/L at each sulphuric acid concentration.

For more details on the investigated system were examined some noticeable works on iron electrodeposition from sulphate electrolytes. For example Mostad et al. were collected the long experience from an industrial plant and semi-plant pilot electrolytic cell and paid attention for using electrode diaphragm for separation of anode side reaction involving iron ions [10]. The authors reported for the important role of solution pH and its role on the current efficiency. At the same time Diaz et al. published polarization curves and impedance diagrams with detailed step increase of the cathode potential and solution pH. After a deep analysis the authors suggest an interesting mechanism of “at least three adsorbed species” of iron electrodeposition from acid sulphate solutions. For more details on the proposed kinetic model see reference [11].

Nevertheless the complicated electrochemical behavior of iron the paper is devoted to the practical importance of ferrous ions on the process of copper recovery. All experiments were carried out from sulphate electrolytes containing cupric ions and ferrous ions in ratios close to the proportion of these two metals in the solid metallurgical wastes, especially secondary precipitates from zinc hydrometallurgy.

## EXPERIMENTAL

Analytical grade iron (II) sulfate heptahydrate, copper (II) sulphate pentahydrate and sulphuric acid were used for electrolyte composition. Ferrous ions (1, 5 or 10 g/L) were added to electrolytes containing 10 or 50 g/L cupric ions in the presence (60 or 130 g/L) and absence of free sulphuric acid (pH=0.1). For metal compounds dissolution and acid dilution laboratory bi-distilled water was used. All chemicals and electrodes gravimetric measurements were carried out on a precise analytical vessel Sartorius BP 301 S.

The potentiodynamic experiments were carried out in a thermostated (37 ± 1$^o$C), three-electrode glass cell (300ml) without stirring of the electrolyte. The cathode (2.0 cm$^2$) and both anodes (4.0 cm$^2$ total area) were Pt plates. The reference electrode was a mercury/mercurous sulphate electrode in 0.5M $H_2SO_4$ (SSE), its potential *vs.* NHE being +0.720 V. The cyclic potentiodynamic studies were carried out by potential scanning at a rate of 30 mV sec$^{-1}$ in the potential range starting from +1.200 to - 1.800 V *vs* SSE using a computerized PAR 263A potentiostat/galvanostat with Soft Corr II software. Galvanostatic depositions were carried out in sulphate electrolytes (500ml) on copper cathodes (5.0 cm$^2$) and Pb-Ag (1%) alloy anodes at a current density of 2 A dm$^{-2}$ for 5 hours.

Current efficiency (CE) in the potentiodynamic regime was determined by graphical integration of obtained cyclic voltammograms (CVA) curves by the help of potentiodynamic software. For more details see next section. In galvanostatic deposition current efficiency was determined by precise gravimetric measurement of the metal quantity deposited on the cathode. Copper plates prepared for working cathode in form were tarred before and after the electrolysis. A special attention was paid when the final deposit was rough and powderly for any losses in the total mass of the working cathode.

To determine the yield at current efficiency is used the relationship between the real gravitate mass of deferred copper coating to the theoretical multiplied by a hundred:

$$CE\ \% = (\triangle m_{cath.} / \triangle m_{theor.})\ .\ 100$$

In the case of cathodic deposition of copper sulphate electrolyte, where copper ions are second valence, $k_e$ accepted values 0,329 mg /C or 1,186 g / A.h relative time one hour:

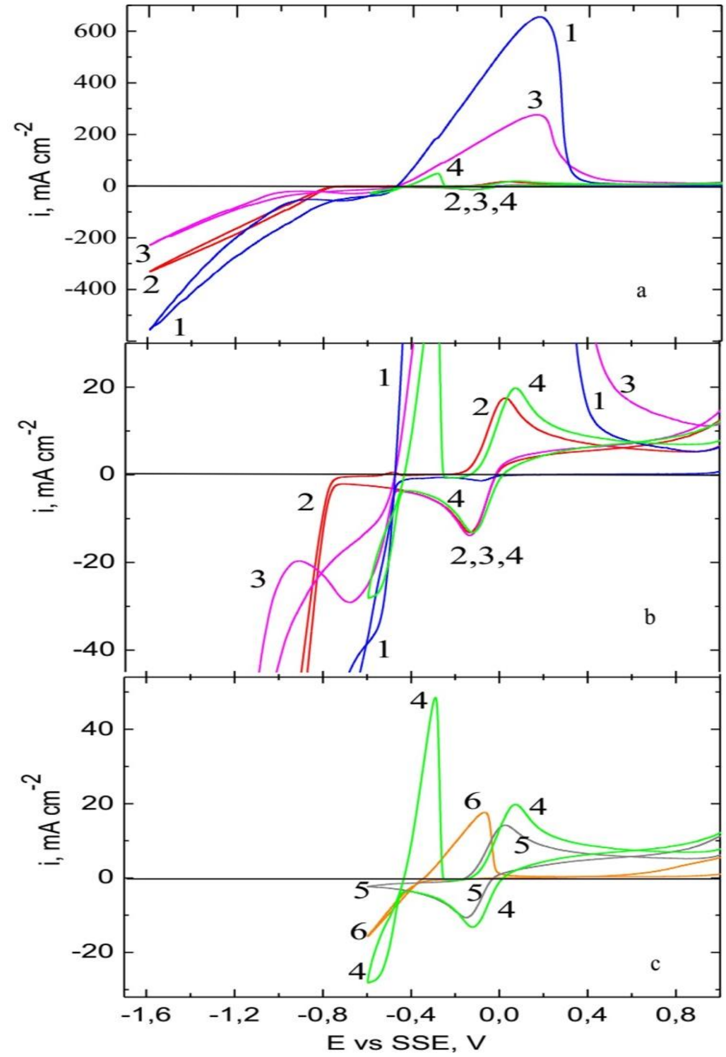
$$\triangle m_{\text{theor.}} \ [g] = 1{,}186\ [g/A.h]\ \ I\ [A]\ \ \triangle t\ [h]$$

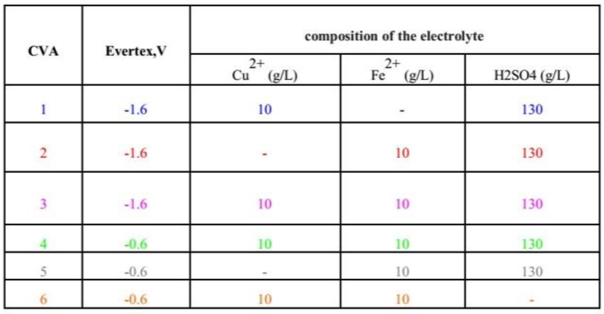
A Philips PW 1050 X-ray powder diffractometer with angular scanning range of 20-110 (2θ) was used. The surface morphology of the deposits was examined and EDX Analysis was performed by scanning electron microscopy (SEM) using a JEOL JSM 6390 microscope.

## RESULTS AND DISCUSSION

### *1. Cyclic voltammetry*

In order to assess the intimate role of ferrous ions in cupric electrolyte potentiodynamic scans were performed. In Figure 1 (a, b, c) by curve 1 was recorded the behavior of copper electrolyte in absence of ferrous ions. The potentials of very first scans were extended to -1.6 V to detect hydrogen evolution area. Potentiodynamic investigations were started with an electrolyte containing 10 g/L cupric ions and 130 g/L sulphuric acid. The electrolyte composition is referred in the auxiliary table in Fig. 1.





| CVA | Evertex,V | composition of the electrolyte | | |
|---|---|---|---|---|
| | | $Cu^{2+}$ (g/L) | $Fe^{2+}$ (g/L) | H2SO4 (g/L) |
| 1 | -1.6 | 10 | - | 130 |
| 2 | -1.6 | - | 10 | 130 |
| 3 | -1.6 | 10 | 10 | 130 |
| 4 | -0.6 | 10 | 10 | 130 |
| 5 | -0.6 | - | 10 | 130 |
| 6 | -0.6 | 10 | 10 | - |

**Fig. 1.** Cyclic voltamperograms of electrolytes containing cupric and ferrous ions: a) view to full negative scan; b) spread area near iron deposition/dissolution; c) restricted scan to $E_{vertex}$ = -0.6 V.

Following the first recorded potential-current trace some important points can be observed. Importantly, at potential -0.400 V the cathodic current starts to increase resulting from the reduction of cupric ions. On further increase in the potential (in negative direction), the cathodic current decreases from -57.1 mA cm$^{-2}$ at -0.745 V to -54.4 mA cm$^{-2}$ at -0.860 V due to the concentration polarization of reaction 1 (Table 1).

**Table 1.** Specific reactions in potentiodynamic regime and standard potentials with reference to a standard hydrogen electrode or a saturated mercury sulphate electrode.

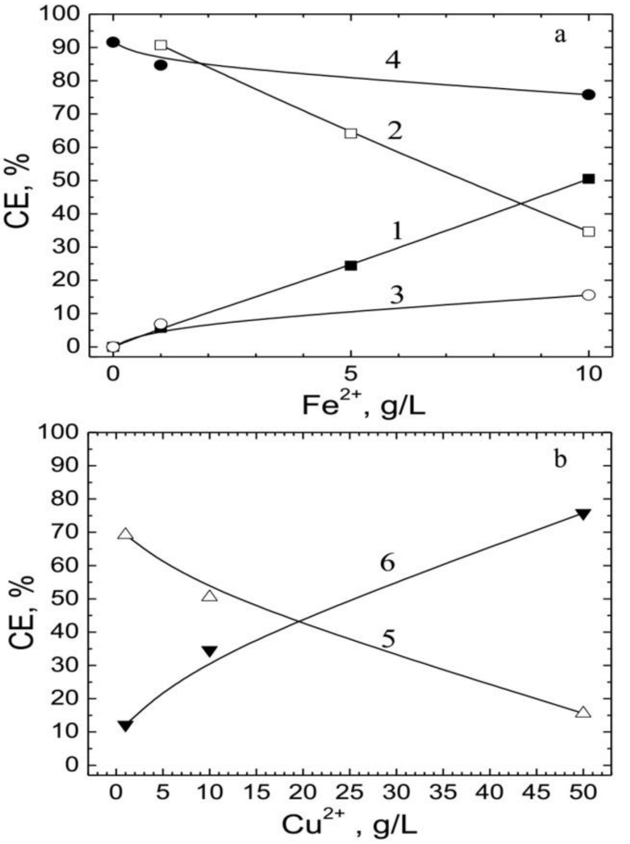
| CVA scanning | | Order of standard potentials | | | |
|---|---|---|---|---|---|
| **Reaction** | **№** | **Half-Reaction** | **№** | ***E vs* NHE, V** | ***E vs* SSE, V** |
| $Cu^{2+} + 2e^{-} = Cu$ | 1 | $Fe^{2+} + 2e^{-} \rightleftarrows Fe$ | 1S | -0.44 | -1.16 |
| $2H^{+} + 2e^{-} = H_2$ | 2 | $Fe^{3+} + 3e^{-} \rightleftarrows Fe$ | 2S | -0.04 | -0.76 |
| $Cu - 2e^{-} = Cu^{2+}$ | 3 | $2H^{+} + 2e^{-} \rightleftarrows H_2$ (g) | 3S | 0.00 | -0.72 |
| $2H_2O - 4e^{-} = 4H^{+} + O_2$ (g) | 4 | $Cu^{2+} + e^{-} \rightleftarrows Cu^{+}$ | 4S | 0.16 | -0.56 |
| $Fe^{3+} + e^{-} = Fe^{2+}$ | 5 | $Cu^{2+} + 2e^{-} \rightleftarrows Cu$ | 5S | 0.34 | -0.38 |
| $Fe^{2+} - e^{-} = Fe^{3+}$ | 6 | $Cu^{+} + e^{-} \rightleftarrows Cu$ | 6S | 0.52 | -0.20 |
| $Fe^{3+} + 3e^{-} = Fe$ | 7 | $Fe^{3+} + e^{-} \rightleftarrows Fe^{2+}$ | 7S | 0.77 | +0.05 |
| $Fe - 3e^{-} = Fe^{3+}$ | 8 | $O_2$ (g) $+ 4H^{+} + 4e^{-} \rightleftarrows 2H_2O$ | 8S | 1.23 | +0.51 |
| $Cu^{2+} + Fe^{2+} = Cu^{+} + Fe^{3+}$ | 9 | | | | |
| $2Cu^{+} = Cu^{2+} + Cu$ | 10 | | | | |

Then the cathodic current begins to increase again as a result of the reactions 1 and 2. During the reverse potential scan (in positive direction), dissolution of the copper coatings starts at -0.470 V (reaction 3) yielding an anodic peak. At +0.900 V, evolution of oxygen commences and the current rises again. Curve 2 shows scan to -1.6 V in an electrolyte containing only 10 g/L ferrous ions and 130 g/L sulphuric acid. At potential -0.020 V, the cathodic current starts to increase resulting from the reduction of ferric ions (reaction 5). The ferric ions are a result of the reaction 6 proceeding on the working or counter electrodes when their potentials are sufficiently positive for the oxidation of ferrous ions to commence. On further increase in potential, the cathodic current decreases due to the concentration polarization of reaction 5. Then (at -0.720 V), the cathodic current begins to increase again as a result of the reactions 2 and 7. During the reverse potential scan dissolution of the iron coating starts at -0.525 V (reaction 8) yielding at -0.485 V very little (0.7 mA cm$^{-2}$) anodic peak. At -0.230V oxidation of ferrous to ferric ions starts and the current rises (reaction 6). At +0.900 V, evolution of oxygen commences and the current rises again as a result of reaction 4.

In the presence of ferrous ions, the reaction 9 proceeds in the bulk electrolyte. Cuprous ions disproportionate rapidly to cupric ions and elemental copper (reaction 10). Since no copper precipitate is observed in the studied electrolytes, it can be concluded that the amount of cuprous ions produced by reaction 9 is very small.

Curves 3 and 4 show scans to -1.6 V or -0.6 V in an electrolyte containing 10 g/L cupric ions, 10 g/L ferrous ions and 130 g/L sulphuric acid. At potential of -0.020 V, the cathodic current starts to increase and at potential -0.130 V the curves feature peaks resulting from the reduction of ferric ions (reaction 5). In this case the ferric ions are as a result both of the oxidation of ferric ions by the reaction 9 taking place in the bulk electrolyte and the reaction 6 proceeding on the working or counter electrodes when their potentials are sufficiently positive for the oxidation of ferrous ions. On further increase in potential, the cathodic current decreases due to the concentration polarization of the reaction 5. At -0.475 V the cathodic current begins to increase again to -29.5 mA cm$^{-2}$ (-0.680 V) as a result of the reactions 1. On further potential increase, the cathodic current decreases to -19.4 mA cm$^{-2}$ (-0.910 V) due to the concentration polarization of the reaction 1. Then the current rises again as a result both of Cu deposition (reaction 1) and hydrogen evolution (reaction 2). During the reverse potential scan, dissolution of the copper coating starts at -0.480 V yielding an anodic peak as a result of reaction 3 with a standard potential of -0.380 V. On further scanning to more positive potentials, the current starts to increase and new, considerably lower anodic peak (curve 4) occur due to the ferrous ions oxidation (reaction 6). At +0.900 V, evolution of oxygen commences (reaction 4) and the current rises.

The graphical integration of the cathodic and anodic peaks of the cyclic voltammograms recorded on scanning to -0.6 V vs. SSE in electrolytes with different composition, allows determination the quantity of electricity ($q_{cath.}$) consumed by the different reactions. Integration of the cathodic peak resulting from reaction 5 and of the anodic peak produced by reaction 3 gives the values of the quantity of electricity consumed for the reduction of ferric ions to ferrous ions ($q_{Fe3+/Fe2+}$) and for the dissolution of copper to cupric ions ($q_{Cu/Cu2+}$), respectively. The remaining part of the cathodic electricity goes for the evolution of hydrogen. This approach is mostly based on the fact that no side chemical reactions proceed during the potential scanning and that no other reactions take place within the integration regions, except for reaction 3 or 5, respectively [12]. The obtained results of the above calculations, which may be called partial contribution (or current efficiency) of the respective reactions to the overall process are presented in Figure 2 a,b.



| № | electrolyte composition | | | E vertex, V |
|---|---|---|---|---|
| | Cu (g/L) | $Fe^{2+}$ (g/L) | $H_2SO_4$ (g/L) | |
| 1 | 10 | 0-10 | 130 | - 0.6 |
| 2 | 10 | 0-10 | | |
| 3 | 50 | 0-10 | | |
| 4 | 50 | 0-10 | | |
| 5 | 0-50 | 10 | | |
| 6 | 0-50 | 10 | | |

**Fig. 2.** Partial contribution of the reactions taking place on the electrodes.

At the lower cupric ion concentration, the part of the reduction of ferric ions increases and even becomes predominating (50.5%) in the electrolyte containing 10 g/L ferrous ions (Fig. 2a, curve 1). The partial contribution of the reduction of cupric ions decreases rapidly with increase of the ferrous ions concentration and becomes 34.6% at 10 g/L ferrous ions (Fig.2a, curve 2). Similar relationships have been observed at 50 g/L cupric ions concentration (Fig. 3a, curves 3 and 4), but in this case the influence of the higher ferrous ions concentration is considerably weaker.

The part of the reduction of ferric ions decreases with the increase of cupric ions concentration (Fig.3 b, curve 5). The partial contribution of the reduction of cupric ions increases rapidly with the increase of cupric ion concentration (Fig. 3 b, curve 6).

### *2. Galvanostatic deposition*

Current efficiency of copper decreases sharply with increase in ferrous ions concentration during deposition in electrolytes containing 10 g/L cupric ions and 130 g/L sulphuric acid (Fig. 3 a) and is practically independent on ferrous ions concentration during deposition in electrolytes containing 50 g/L cupric ions and 130 g/L sulphuric acid (Fig. 3 b). When the deposition is conducted in electrolytes containing 50 g/L cupric ions and 130 g/L sulphuric acid, the current efficiency is higher than 90%. The very weak influence of ferrous ions on the recovery of copper from electrolytes containing 50 g/L cupric ions can be explained by the impeded access to the cathode of the ferric ions formed on the anodes. The reduction of ferric ions to ferrous ions on the cathode and increased hydrogen gas evolution may lead to a decrease in copper current efficiency. This is, probably, the reason for the significantly lower current efficiency when the concentrations of cupric ions and ferrous ions are equal (10 g/L).

The results of the microprobe analysis indicate that obtained metal coatings contains only copper phase (Fig. 4 a-d). However, in the presence of ferrous ions the coatings have a more fine-grain structure. The coatings are dark red, fragile and brittle (powdery).

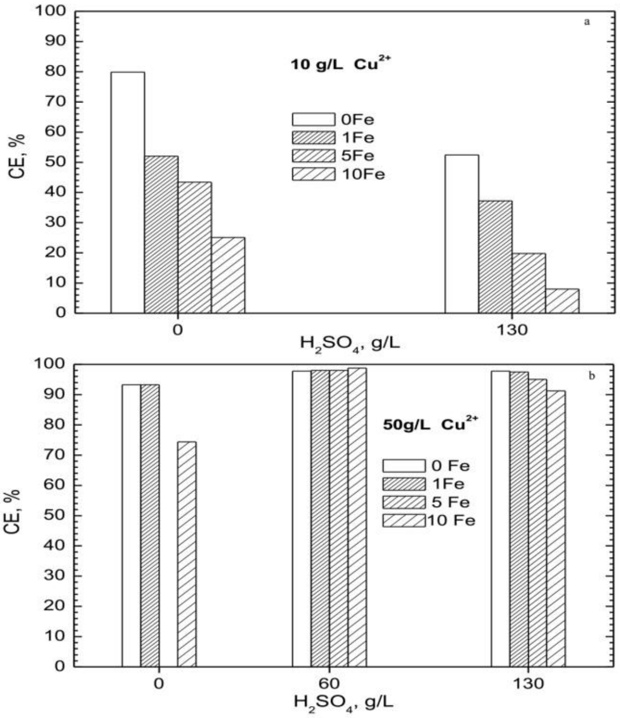


**Fig. 3.** Current efficiency of copper depositions depending on ferrous ion concentration and the presence of free sulfuric acid. Deposition at current density 2.0 A/dm$^2$ for 5 hours.

In structural aspect it is seen that the preferred orientation of the Cu coating changes from a plane of lower density (220) (Fig. 5 a) to a plane of higher density (111) (Fig.5 b) despite the fact that EDX results show that no iron is co-deposited with the copper. However, the EDX technique is not sufficiently accurate to detect small quantities of metals due to its poor precision. A more detailed information can be obtained if the value of cell parameter is calculated from the X-ray diffraction data. If small quantities of ferrous ions are incorporated in the cell, it would provoke a distortion in the copper cell parameter. The formation of alloys, even in small portion, could also explain the change in the preferred orientation observed in the copper deposit. It is possible also iron ions to inhibit the growth of crystal face with (220) orientation.

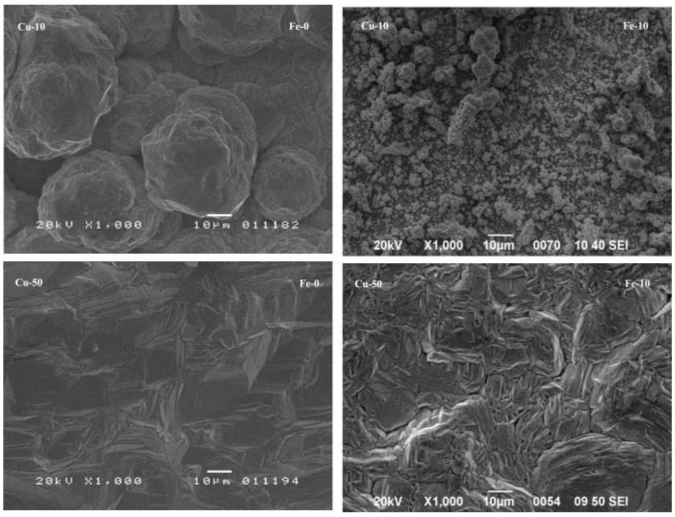


**Fig. 4.** Morphology of deposited copper coatings in the presence and absence of ferrous ions**.** SEM images x1000, current density 2.0 A/dm$^2$, deposition time 5.0 h, free sulphuric acid 130 g/L. Metal ion concentrations shown on pictures in g/L.

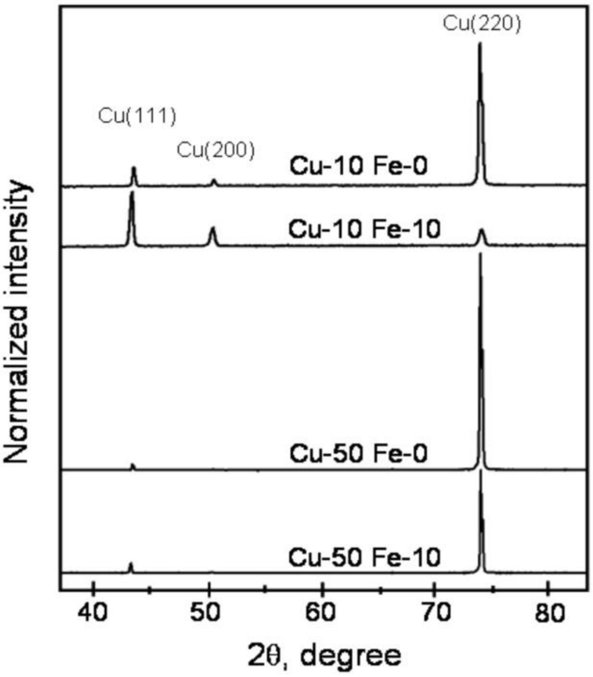


**Fig. 5.** Powder diffraction analyses of copper samples.

The surface morphology of coatings is not changed in the presence of ferrous ions when cupric ions dominate in the solution (Fig. 5 c, d). At similar ratio between cupric and ferrous ions the microstructure is changed substantially (Fig. 5 a, b). In both cases the obtained coatings are light red in color, smooth and semi-bright. The SEM microprobe analysis detects only copper content in all tested samples (Fig. 4 a-d).

The results of X-ray diffraction analysis of coatings deposited in acidic electrolytes in the absence of ferrous ions (Fig. 5 a, c) and in the presence of ferrous ions (Fig. 5 b, d) shows that at higher (50 g/L) cupric ions concentration the preferred (220) orientation of the coatings is not changed which can be explained by the impeded access to the cathode of the ferric ions formed on the anodes.

The ferrous ions have a similar potential to that of copper ions. This circumstance makes it almost impossible their electrochemical separation, although the influence of the sulphuric acid the two metals have separate, distinct peaks anodic dissolution. In cathodic reduction, however, cupric and ferrous ions practically tend to a codeposition process. Therefore, attention to ferrous ions is mainly directed to their role on the morphology and purity of the resulting coatings and the extraction stream.

## CONCLUSIONS

Using classical electrochemical methods as cyclic voltamperometry and galvanostatic deposition was demonstrated the contribution of ferrous ions on the behavior of copper sulphate electrolytes. The obtained results were aimed for application to real systems by selective electroextraction of valuable non-ferrous metals from secondary wastes. By scanning of the electrode potential were selected the specific conditions for best observation of separate and mixed copper and iron metallic phases. The presence of free sulfuric acid affirmatively contributes to copper-iron separation and minimization of their co-deposition. At comparatively large amounts of ferrous ions the observed reduction of current efficiency was overcome by simply increasing the cupric part. In galvanostatic regime this allows practical recovery of copper metal despite the presence of iron contaminants with low electricity losses and sufficient purity. In this study ferrous ions have a significant influence in cases where the copper ions are at a concentration up to 10 g / L, the current efficiency by reducing of the copper ions of less than 40%. At high concentrations of copper ions in the electrolyte, in the range of 50 g/ L, the presence of ferrous ions decreases the current efficiency to a level not lower than 90%. This reduction occurs at concentrations comparable to those of copper ions (10 g/L). In this case, the impurity ferrous ions will mostly have a role on the morphology and purity of the resulting copper coatings without compromising recovery as a whole. In all cases where the content of minded copper is low and in the same time ferrous ions in the waste electrolyte is around and above 1.0 g/L it is appropriate that they should be removed before conducting selective electrochemical recovery.

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# ЕЛЕКТРОХИМИЧНО ИЗВЛИЧАНЕ НА МЕД В ПРИСЪСТВИЕТО НА ВРЕДНИ ЗА ПРОЦЕСА ФЕРОЙОНИ

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(Резюме)

Целта на това изследване е да се съберат конкретни електрохимични условия за разделно отлагане на мед от смесени медно-железни моделни електролити. Изследването е посветено за по-нататъшно използване на богати медни индустриални отпадъци. Съставът на изследваните електролити е съобразен с реалните твърди и излужени вторични отпадъци. Чрез потенциодинамични и галваностатични методи е осъществена електро-екстракцията на мед в присъствието на железни йони от кисели сулфатни електролити. Потенциодинамичното поведение на медта е тествано чрез добавяне на феройони към изследваната система. Получените експериментални резултати показват, че вредното въздействие на феройоните върху отлагането на медта може да бъде сведено до минимум чрез увеличаване на медната компонента. Железните йони могат да бъдат разделени от медните йони в потенциодинамичен режим чрез ограничаване на обхвата на потенциала на сканиране. Във всички случаи, наличието на железни йони намалява анодните пикове на разтваряне на медта. В галваностатичен режим се наблюдава рязко намаляване на добива по ток с увеличаване концентрация на феро-йоните. При висока концентрация на медните йони този важен параметър е по-висок от 90% и на практика е независим от присъствието на железни йони. Получените медни покрития от еднакво смесени мед-желязо електролити са тъмно червени на цвят, рехави и прахообразни. При по-високо съотношение на мед-желязо 5: 1, покритията са светло червени на цвят, гладки и блестящи. От проведените СЕМ и прахово-дифракционен анализ се вижда, че покритията съдържат единствено метална мед.