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# Determination of Cu<sup>2+</sup> Ions Concentration by Electrochemical Methods

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## Abstract

The possibility of determining the concentration of heavy metal ions, *i.e.*, Cu<sup>2+</sup> ions in an aqueous solution of CuSO<sub>4</sub> · 5H<sub>2</sub>O, was tested by electrochemical means. For this purpose, polarisation curves were recorded on a Pt electrode in solutions of known (0.1, 0.5, 1.0, 2.5, 5.0, 7.5, 10.0, 50.0, and 100.0 mM) and unknown concentrations of Cu<sup>2+</sup> ions with the reading of the limiting diffusion current. Polarisation measurements were performed in the potential range from open circuit potential to –3.0 V with different potential scan rates (1, 5, 10, 20, and 50 mV s<sup>–1</sup>). By cathodic deposition on the Pt surface, a red-dish-brown copper coating was deposited, the thickness of which increased as the concentration of Cu<sup>2+</sup> ions in the solution increased. Furthermore, the limiting diffusion current *i*<sub>d</sub> increased linearly with the increase in Cu<sup>2+</sup> ion concentration, *c*. The *i*<sub>d</sub> = *f*(*c*) plot was constructed and the unknown concentrations of Cu<sup>2+</sup> ions in three randomly prepared CuSO<sub>4</sub> · 5H<sub>2</sub>O solutions were determined. Heavy metal ions from polluted waters (Cu<sup>2+</sup> ions) can be successfully removed by electrochemical means, and their concentration determined.

## Keywords

Heavy metal ions, copper, polarisation measurements, electrodeposition, limiting diffusion current

## 1 Introduction

The increases in production capacity in all sectors of industry and the development of new products using modern technologies have led to the problem of environmental pollution. Water, air, and soil are polluted with toxic and carcinogenic substances of inorganic and organic origin.

Environmental protection strategies in the industry generally include waste treatment processes, and the development of new processes that have no harmful effects on the environment.<sup>1–3</sup> Electrochemistry plays an important role in both of these strategic goals. In this sense, electrochemical processes and devices for the purification of wastewater, flue gases, contaminated groundwater and soil are addressed, as well as ways to improve existing electrochemical processes or products.<sup>4–15</sup>

A large and important area of environmental concern is in the field of energy production and transport. Electrochemistry offers a unique opportunity to generate clean energy with high utilisation rates in fuel cells or its storage in ecological batteries. Such energy sources can be used in higher-power stationary plants or in electric vehicles.<sup>5–7</sup> The potential of electrochemistry for environmental protection has been explored in numerous review articles and monographs.<sup>4–16</sup> The first book devoted to this topic was published in 1972.<sup>4</sup> Many of the concepts of electrochemical environmental protection presented in this publication have been applied in practice.

Electrochemical technologies in wastewater and drinking water treatment were expensive methods in the past. However, advances in the development of electrode materials and electrochemical technology offer additional new, more effective, and economical solutions in this area of environmental protection.<sup>9–13</sup> Electrochemical water purification technologies include: electrodeposition, electrocoagulation, electroflotation, and electrochemical oxidation.<sup>6,12,13</sup>

Wastewaters from electrochemical industries (electroplating, metallurgy, metal processing, printing plate and battery manufacturing) are contaminated with metal ions and require special treatment to remove them or convert them into useful material. Traditional purification of such waters uses the hydroxide precipitation method, which produces an extensive sludge of metal hydroxide that must be disposed of in an environmentally safe manner. If the water is contaminated with heavy metal ion complexes, the hydroxide precipitation method is not acceptable.<sup>12</sup>

The electrochemical process for the extraction of heavy metals represents a simple alternative approach, as metal ions from wastewater can be converted into pure metal form. The process consists of a simple reaction of reducing metal ions to their elemental state at the cathode.<sup>12,17,18</sup>

This work investigated the possibility of removing heavy metal ions, especially Cu<sup>2+</sup> ions, from polluted waters, and determining their concentration by electrochemical means.

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## 2 Experimental

Electrochemical measurements were performed using a Princeton Applied Research PAR 273A potentiostat/galvanostat in conjunction with a standard double-walled glass

cell with three electrodes: Pt sheet working electrode, Pt spiral wire counter electrode, and saturated calomel reference electrode (SCE).

The working electrode was a Pt sheet with a large surface area (15 cm<sup>2</sup>), which allowed easier deposition of Cu. The contact was made by soldering an insulated Cu wire to a Pt sheet. The made contact was then insulated with epoxy resin. Before each measurement, the Pt electrode was chemically cleaned in an HNO<sub>3</sub> solution (1 : 1) for 30 s. The electrode was then washed with distilled and redistilled water, and placed in the electrochemical cell.

A 1 M CuSO<sub>4</sub> · 5H<sub>2</sub>O solution was used as the basic electrolyte, prepared by dissolving 249.6 g of the solid salt CuSO<sub>4</sub> · 5H<sub>2</sub>O (p.a.) in redistilled water to a final volume of 1000 ml. By diluting this solution with redistilled water, solutions with different but known concentrations *c* (0.1, 0.5, 1.0, 2.5, 5.0, 7.5, 10.0, 50.0, and 100.0 mM) were prepared. In addition, three Cu<sup>2+</sup> solutions of unknown concentration were prepared by random dilution of the basic electrolyte; *c*<sub>1x</sub>, *c*<sub>2x</sub>, *c*<sub>3x</sub>. All measurements were performed at a temperature of 20 °C. During thermostating and performing measurements, the electrolyte solution was mixed continuously with a magnetic stirrer at a constant rotation rate of 120 rpm.

The potentiodynamic polarisation method enabled measurement of the limiting diffusion current *i*<sub>d</sub>, which is directly proportional to the concentration of electroactive ions in the solution (in this case Cu<sup>2+</sup> ion). In this method, the potential of the working electrode, *E*, was changed in the cathodic direction (at a certain rate), and the change in current density, *i*, which is a consequence of the reduction of Cu<sup>2+</sup> ions at the Pt electrode, was monitored.

At the beginning of the measurement, a solution with the lowest concentration of Cu<sup>2+</sup> ions (0.1 mM) was poured into the electrochemical cell, and was thermostatted and stirred. Meanwhile, the working electrode (Pt sheet) was chemically cleaned in HNO<sub>3</sub> solution, washed with distilled and redistilled water, and inserted into the electrochemical cell.

The polarisation measurement was carried out in the potential range from open circuit potential (*E*<sub>OC</sub>) to –3.0 V with a scan rate of 10 mV s<sup>–1</sup>. At the end of the measurement, the platinum electrode (with deposited Cu layer) was washed with water, and photographed. The procedure was repeated for all prepared solutions with a known concentration of Cu<sup>2+</sup> ions (0.5, 1.0, 2.5, 5.0, 7.5, 10.0, 50.0, and 100.0 mM), and for solutions with an unknown concentration of Cu<sup>2+</sup> ions (*c*<sub>1x</sub>, *c*<sub>2x</sub>, *c*<sub>3x</sub>). From the polarisation curves, the limiting diffusion currents were determined for solutions with known (*i*<sub>d</sub>) and unknown (*i*<sub>d</sub><sub>x</sub>) concentrations of Cu<sup>2+</sup> ions.

In addition, measurements were performed to determine the influence of the scan rate on the reduction reaction of the copper ions. For this purpose, polarisation curves were recorded in a 5.0 mM CuSO<sub>4</sub> · 5H<sub>2</sub>O solution in the potential range from *E*<sub>OC</sub> to –3.0 V with scan rates of 1, 5, 10, 20, and 50 mV s<sup>–1</sup>.

### 3 Results and discussion

Wastewaters from electrochemical industries (electroplating, metallurgy, metal processing, printed circuit boards, and battery manufacturing) are contaminated with metal ions and require special treatment to remove them or convert them into useful material. Traditional purification of such waters uses the hydroxide precipitation method, which produces an extensive sludge of metal hydroxide that must be disposed of in an environmentally safe manner. If the water is contaminated with heavy metal ion complexes, the hydroxide precipitation method is not acceptable.<sup>12,13</sup>

The electrochemical process for heavy metal extraction is a simple alternative approach because metal ions from wastewater can be converted to the pure metal form. The process consists of a simple reduction of metal ions to their elemental state on a suitable cathode material:



Although the process is basically simple, it encounters numerous problems and limitations. The basic problem is mass transfer, *i.e.*, the transfer of metal ions from the bulk solution to the cathode surface, which is limited by the establishment of a limiting diffusion current, *i*<sub>d</sub>.<sup>12</sup>

The limiting diffusion current, and thus, the speed of the metal deposition process in dilute solutions, are very low. To increase the speed of the process, the electrode surface area must be increased, or the mass transfer must be increased in some way, which increases the dimensions and the general investment costs for reactor construction.<sup>12</sup>

In accordance with thermodynamic predictions, it is possible to reduce the concentration of metal ions in the bulk of the solution to a relatively low value if the electrode potential is more negative than the standard potential of the redox pair Me<sup>z+</sup>/Me. However, as mentioned previously, reducing the concentration of the solution significantly reduces the speed of the process controlled by the transfer of substances (limiting current). In practice, this means that electrolysis at concentrations of less than 0.05 ppm takes too long and becomes uneconomical.<sup>12</sup>

If we consider the cathodic deposition process of the metal, the reaction rate *v*<sub>er</sub> (the amount of reacted substance per unit time and area), *i.e.*, the cathodic current density, *i*<sub>k</sub>, is compensated by the amount coming from the solution mass in the same time, *i.e.*, it is compensated by the corresponding diffusion flux *J*<sub>d</sub>.<sup>19–21</sup> In the steady state, these two influences balance each other out, which can be described by the following equation:

$$\frac{i_k}{zF} = D \frac{c_o - c_e}{\delta} \quad (2)$$

If we rearrange Eq. (2) and express it explicitly by *i*<sub>k</sub>, we obtain:

$$i_k = \frac{DzF}{\delta} (c_o - c_e) \quad (3)$$

In Eqs. (2) and (3),  $i_k$  is the cathode current density (rate of metal deposition on the cathode),  $c_e$  is the local concentration of ions on the electrode surface,  $c_o$  is the concentration of ions in the bulk of the solution,  $\delta$  is the thickness of the diffusion layer,  $D$  is the diffusion coefficient,  $z$  is the number of electrons exchanged, and  $F$  is Faraday's constant. Eq. (3) clearly shows that we can increase the current density in the cell and thus decrease the local concentration. We can only achieve this increase in current density up to a certain limit when the local concentration becomes zero. A further increase in the cathodic polarisation of the electrode does not lead to a corresponding increase in the current, and the so-called "current plateau" phenomenon is observed on the polarisation curve. This fixed maximum value of the current is called the limiting diffusion current ( $i_d$ ), and is determined by the following relationship:

$$i_d = \frac{DzF}{\delta} c_o \quad (4)$$

With a constant thickness of the diffusion layer and a constant temperature, the magnitude of the  $i_d$  is directly proportional to the concentration of ions in the solution:<sup>19-21</sup>

$$i_d = kc_o \quad (5)$$

Since the measurements were performed at a constant temperature (20 °C) and constant electrolyte stirring (magnetic stirrer; 120 rpm), it can be considered that the diffusion layer had reached a "quasi-constant" thickness, and that satisfactory conditions had been reached for the application of Eq. (5).

The limit value of the  $i_d$  thus represents the maximum rate at which the deposition process can take place. This dependence plays a very important role for electroanalytical purposes in determining the unknown concentration of the respective metal cation in the solution.

In this work, the electrochemical process of removing Cu<sup>2+</sup> ions from an aqueous solution of CuSO<sub>4</sub> · 5H<sub>2</sub>O was simulated. The aim of the measurement was to determine an unknown concentration of Cu<sup>2+</sup> ions in the solution by recording potentiodynamic polarisation curves ( $i$ - $E$  dependencies) in solutions with known concentrations of Cu<sup>2+</sup> ions and reading the  $i_d$ .

Fig. 1 shows the cathodic polarisation curve recorded at a Pt electrode in a 1.0 mM CuSO<sub>4</sub> · 5H<sub>2</sub>O solution. When the potential was changed in the cathodic direction, an increase in current was observed due to the acceleration of the Cu deposition reaction at the Pt cathode, which decreased the concentration of Cu<sup>2+</sup> ions near the electrode. However, as cathodic polarisation (and time) depleted the solution layer near the Pt electrode of electroactive species (*i.e.*, Cu<sup>2+</sup> ions), the current increased more and more slowly. When the concentration of Cu<sup>2+</sup> ions on the electrode surface dropped to zero, a limiting diffusion current was established, and a so-called "current plateau" or independence of the current from the potential (in Fig. 1, this corresponds to the potential range from  $\approx -1.2$  V to  $\approx -2.0$  V). Although the cathodic potential increased in this range, the current density (*i.e.*, the reaction rate) re-

mained the same. At sufficiently high cathodic potentials, other electrode processes can also take place (*e.g.*, hydrogen excretion), and the current increases again.

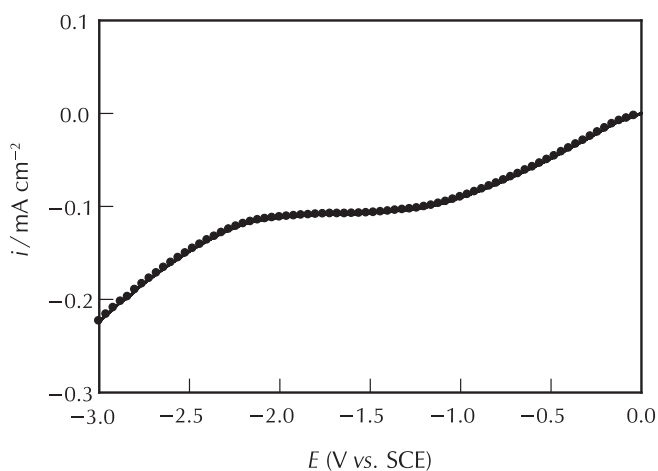


Fig. 1 – Cathodic polarisation curve on Pt electrode in a 1.0 mM CuSO<sub>4</sub> · 5H<sub>2</sub>O solution ( $v = 10 \text{ mV s}^{-1}$ )

Slika 1 – Katodna polarizacijska krivulja na Pt elektrodi u 1,0 mM otopini CuSO<sub>4</sub> · 5H<sub>2</sub>O ( $v = 10 \text{ mV s}^{-1}$ )

Fig. 2 shows polarisation curves recorded in a CuSO<sub>4</sub> · 5H<sub>2</sub>O solution of different concentrations with a constant potential scan rate. With increasing Cu<sup>2+</sup> ion concentration, the current plateau decreased towards more negative values, the limiting cathodic diffusion current became larger (Table 1). Also, with increasing Cu<sup>2+</sup> ion concentration the potential range in which the current plateau occurred became narrower, and in the case of concentrations above 10.0 mM, the problem occurred in determining the region of linear currents (and  $i_d$ ). Accordingly, with the  $i$ - $E$  dependence recorded at the highest examined concentration of Cu<sup>2+</sup> ions (*i.e.*, at 100.0 mM), it was not possible to determine  $i_d$ . The problem could, most likely, be solved by reducing the scan rate or increasing the stirring speed.

Table 1 – Values of  $i_d$  for Pt in CuSO<sub>4</sub> · 5H<sub>2</sub>O solutions of different concentrations ( $v = 10 \text{ mV s}^{-1}$ )

Tablica 1 – Vrijednosti  $i_d$  za Pt u otopinama CuSO<sub>4</sub> · 5H<sub>2</sub>O različitih koncentracija ( $v = 10 \text{ mV s}^{-1}$ )

C / mM	$ i_d  / \text{mA cm}^{-2}$
0.1	0.02
0.5	0.06
1.0	0.11
2.5	0.24
5.0	0.45
7.5	0.69
10.0	0.93
50.0	4.55

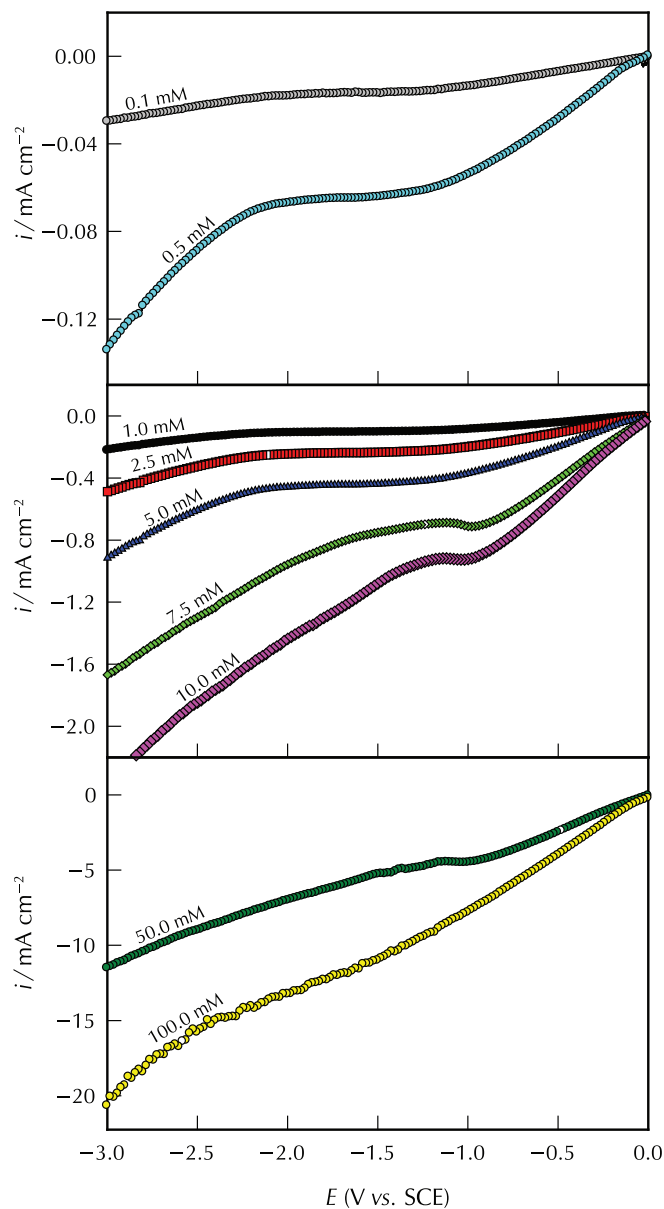


Fig. 2 – Cathodic polarisation curves on Pt electrode in  $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$  solutions of different concentrations ( $v = 10 \text{ mV s}^{-1}$ )

Slika 2 – Katodne polarizacijske krivulje na Pt elektrodi u otopinama  $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$  različitih koncentracija ( $v = 10 \text{ mV s}^{-1}$ )

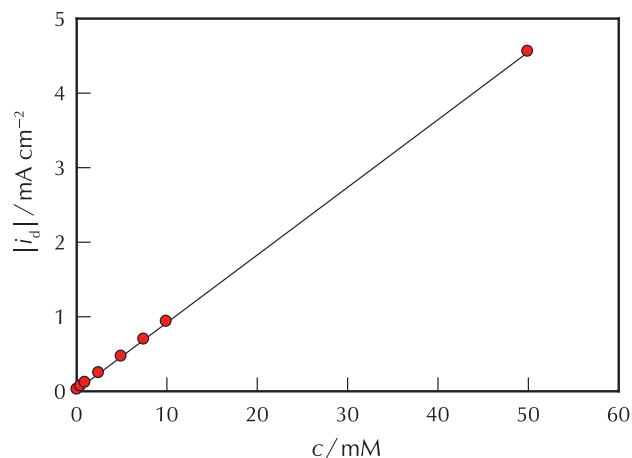


Fig. 3 – Dependence of the limiting diffusion current on the  $\text{Cu}^{2+}$  ion concentration ( $v = 10 \text{ mV s}^{-1}$ )

Slika 3 – Ovisnost granične difuzijske struje o koncentraciji  $\text{Cu}^{2+}$  iona ( $v = 10 \text{ mV s}^{-1}$ )

Based on the data obtained, a diagram of the dependence of the limiting diffusion current on the  $\text{Cu}^{2+}$  ion concentration,  $i_d = f(c)$ , was drawn up, as shown in Fig. 3.

As may be seen,  $i_d$  increased with increasing electrolyte concentration. Furthermore, an extremely linear dependence of the limiting diffusion current on the  $\text{Cu}^{2+}$  ion concentration may be observed, which is consistent with Eq. (5).

At the end of the electrolysis process, the surface of the Pt electrode was photographed with a deposited Cu layer from solutions of different concentrations, and the results are shown in Fig. 4. It may be seen that a thicker layer of reddish-brown copper formed on the Pt surface at higher electrolyte concentrations.

Fig. 5 shows the polarisation curves recorded in  $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$  solutions with an unknown concentration of  $\text{Cu}^{2+}$  ions. For unknown concentrations  $c_{1x}$ ,  $c_{2x}$ , and  $c_{3x}$ , the following limiting diffusion currents were determined: 0.29, 0.56, and 0.82  $\text{mA cm}^{-2}$ .



Fig. 4 – Pt electrode with deposited Cu layer from  $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$  solutions of different concentrations: a) 1.0, b) 2.5, c) 5.0, d) 7.5, and e) 10.0 mM

Slika 4 – Pt elektroda s istaloženim slojem Cu iz otopina  $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$  različitih koncentracija: a) 1,0, b) 2,5, c) 5,0, d) 7,5 i e) 10,0 mM



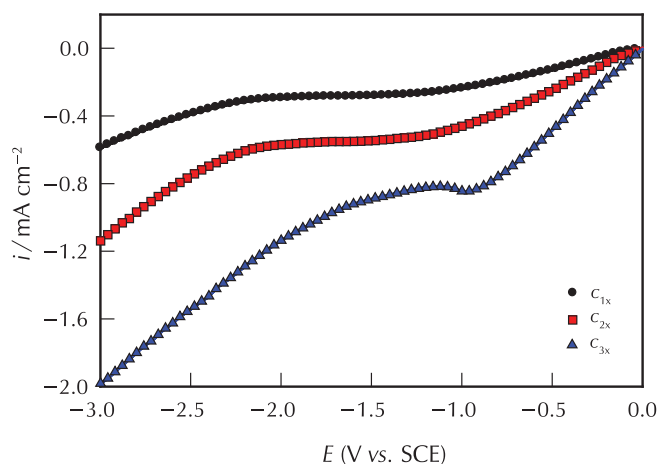


Fig. 5 – Cathodic polarisation curves on Pt electrode in  $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$  solutions of unknown concentrations ( $\nu = 10 \text{ mV s}^{-1}$ )

Slika 5 – Katodne polarizacijske krivulje na Pt elektrodi u otopinama  $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$  nepoznatih koncentracija ( $\nu = 10 \text{ mV s}^{-1}$ )

Using linear  $i_d$ - $c$  dependence (Fig. 3), the limiting diffusion currents ( $0.29$ ,  $0.56$ , and  $0.82 \text{ mA cm}^{-2}$ ) are related to the corresponding  $\text{Cu}^{2+}$  ion concentration, as shown in Fig. 6. The unknown concentrations of  $\text{Cu}^{2+}$  ion were, respectively:  $3.0$ ,  $6.0$ , and  $9.0 \text{ mM}$  (the stated values were confirmed with a spectrophotometer).

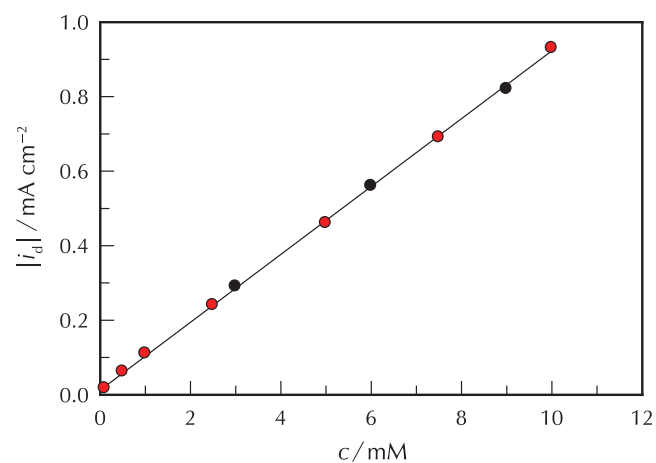


Fig. 6 – Determination of the unknown concentrations of  $\text{Cu}^{2+}$  ions from known values of  $i_d$

Slika 6 – Određivanje nepoznate koncentracije  $\text{Cu}^{2+}$  iona iz poznatih vrijednosti  $i_d$

Part of the measurements was carried out to determine the influence of the scan rate on the copper ion reduction reaction. Thus, Fig. 7 shows polarisation curves recorded in a  $5.0 \text{ mM CuSO}_4 \cdot 5\text{H}_2\text{O}$  solution at potential scan rates ( $\nu = 1, 5, 10, 20$ , and  $50 \text{ mV s}^{-1}$ ). As the scan rate increased, the potential region for copper deposition (i.e., the potential where the limiting diffusion currents

form) became narrower, and moved towards more positive potential values. It may also be observed that the limiting diffusion current increased slightly with the increasing speed of potential scan rate (Fig. 8).

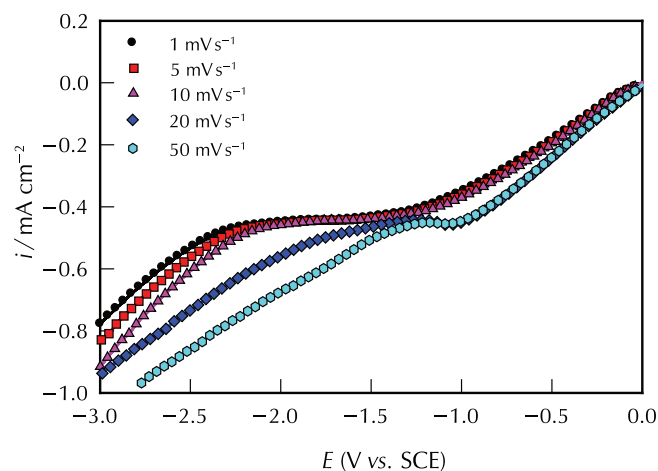


Fig. 7 – Cathodic polarisation curves on Pt electrode in  $5.0 \text{ mM CuSO}_4 \cdot 5\text{H}_2\text{O}$  solution at different potential scan rates

Slika 7 – Katodne polarizacijske krivulje na Pt elektrodi u  $5,0 \text{ mM}$  otopini  $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$  kod različitih brzina promjene potencijala

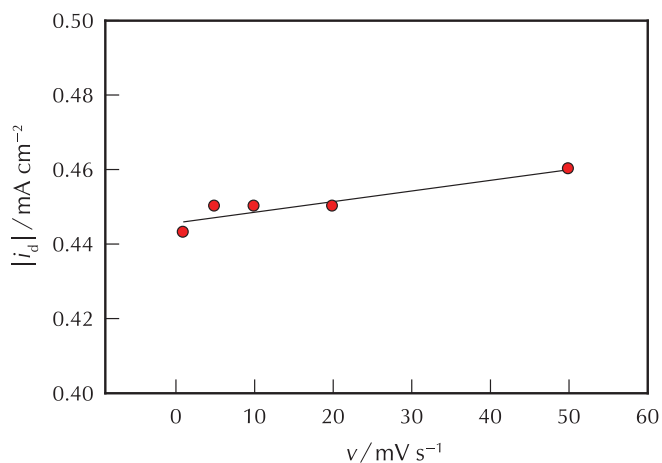


Fig. 8 – Dependence of limiting diffusion current on potential scan rate in  $5.0 \text{ mM CuSO}_4 \cdot 5\text{H}_2\text{O}$  solution

Slika 8 – Ovisnost granične difuzijske struje o brzina promjene potencijala u  $5,0 \text{ mM}$  otopini  $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$

The listed observations additionally confirm the fact that the  $\text{Cu}^{2+}$  ion deposition process is under diffusion control.

## 4 Conclusion

When the potential was changed in the cathodic direction, an increase in the cathodic current was observed due to the acceleration of the electrodeposition of  $\text{Cu}^{2+}$  ions

at the Pt electrode, while the concentration of Cu<sup>2+</sup> ions near the electrode decreased. However, when the cathodic polarisation (and with time) depleted the solution layer near the Pt electrode of Cu<sup>2+</sup> ions, the cathodic current increased more and more slowly. When the surface concentration of Cu<sup>2+</sup> ions approached zero, a limiting diffusion current ( $i_d$ ) was established, and the so-called “current plateau” on polarisation curve had formed.

- The  $i_d$  increased linearly with the increase in Cu<sup>2+</sup> concentration (in range from 0.1 to 50 mM), which corresponds to the equation  $i_d = kc$ .
- The diagram  $i_d = f(c)$  was constructed.
- Using the diagram  $i_d = f(c)$ , the concentrations for three unknown solutions of Cu<sup>2+</sup> ions were determined.
- At higher Cu<sup>2+</sup> concentrations, a thicker layer of red-dish-brown copper coating formed on the Pt surface.
- The process of Cu<sup>2+</sup> ion deposition is subject to diffusion control.
- The Cu<sup>2+</sup> concentrations can be determined successfully and simply by electrochemical methods.

#### List of abbreviations and symbols Popis kratica i simbola

- $E$  – potential, V  
– potencijal, V
- $v$  – potential scan rate, mV s<sup>-1</sup>  
– brzina promjene potencijala, mV s<sup>-1</sup>
- $i$  – current density, mA cm<sup>-2</sup>  
– gustoća struje, mA cm<sup>-2</sup>
- $i_k$  – cathodic current density, mA cm<sup>-2</sup>  
– katodna gustoća struje, mA cm<sup>-2</sup>
- $i_d$  – limiting diffusion current, mA cm<sup>-2</sup>  
– granična difuzijska struja, mA cm<sup>-2</sup>
- $c$  – concentration, mM  
– koncentracija, mM
- $c_e$  – local concentration on electrode surface, mol dm<sup>-3</sup>  
– lokalna koncentracija na površini elektrode, mol dm<sup>-3</sup>
- $c_o$  – concentration in the bulk of the solution, mol dm<sup>-3</sup>  
– koncentracija u masi otopine, mol dm<sup>-3</sup>
- $D$  – diffusion coefficient, m<sup>2</sup> s<sup>-1</sup>  
– koeficijent difuzije, m<sup>2</sup> s<sup>-1</sup>
- $\delta$  – thickness of the diffusion layer,  $\mu$ m  
– debljina difuzijskog sloja,  $\mu$ m
- $z$  – number of exchange electrons  
– broj izmijenjenih elektrona
- $F$  – Faraday’s constant, 96 488 C mol<sup>-1</sup>  
– Faradajeva konstanta, 96 488 C mol<sup>-1</sup>

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## SAŽETAK

### Određivanje koncentracije Cu<sup>2+</sup> iona elektrokemijskim metodama

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Elektrokemijskim putem ispitana je mogućnost određivanja koncentracije iona teških metala, odnosno Cu<sup>2+</sup> iona u vodenoj otopini CuSO<sub>4</sub> · 5H<sub>2</sub>O. U tu svrhu su na elektrodi od Pt snimane polarizacijske krivulje u otopinama poznatih (0,1, 0,5, 1,0, 2,5, 5,0, 7,5, 10,0, 50,0, i 100,0 mM) i nepoznatih koncentracija Cu<sup>2+</sup> iona uz očitavanje granične difuzijske struje. Polarizacijska mjerenja provedena su u području potencijala od potencijala otvorenog strujnog kruga do –3,0 V uz različite brzine promjene potencijala (1, 5, 10, 20, i 50 mV s<sup>-1</sup>). Katodnom depozicijom na površini Pt taloži se prevlaka bakra crvenkasto-smeđe boje čija je debljina veća što je koncentracija Cu<sup>2+</sup> iona u otopini veća. Nadalje, granična difuzijska struja, *i<sub>d</sub>*, linearno raste s porastom koncentracije Cu<sup>2+</sup> iona, *c*. Konstruiran je *i<sub>d</sub>* = *f*(*c*) dijagram te su određene nepoznate koncentracije Cu<sup>2+</sup> iona u tri nasumično pripremljene otopine CuSO<sub>4</sub> · 5H<sub>2</sub>O. Elektrokemijskim putem uspješno se mogu ukloniti ioni teških metala iz onečišćenih voda (Cu<sup>2+</sup> ioni) te odrediti njihova koncentracija.

#### Ključne riječi

Ioni teških metala, bakar, polarizacijska mjerenja, elektrotaloženje, granična difuzijska struja

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