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Bangladesh J. Sci. Ind. Res. **55(3)**, 229-236, 2020

Short Communication

BANGLADESH JOURNAL OF SCIENTIFIC AND INDUSTRIAL RESEARCH

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Electrodeposition behaviour of copper from ore leachate and copper ammonium sulphate

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Abstract

The deposition behaviour of copper ammonia complexes from ore leachate and synthetic copper ammonium sulphate solutions was investigated using cyclic voltammetrywith platinumas counter electrode. The work is carried out to understand the deposition behaviour of the ore for hydrometallurgical and electro winning application. The chemical reduction and deposition of copper from both solutions consisted of two reversible electrochemical processes, each involving the transfer of a single electron. The $Cu(NH_3)_4^{2+}$ complex in the copper leachate is first reduced to $Cu(NH_3)_4^{+}$ before being reduced to metallic copper. With synthetic copper ammonium sulphate ($Cu(NH_3)_4SO_4$), the reduction to metallic copper is a ligand-coupled electron transfer reaction which proceeds as two sequential, single-electron transfer processes. The $Cu/Cu(NH_3)_4^{2+}$ redox reaction during deposition of copper from the leachate is fast compared to that of the Cu/Cu^{2+} redox reaction in the $Cu(NH_3)_4SO_4$ synthetic solution. Investigation of the electrochemical kinetics shows that the linear relationship between the peak current and the square root of the scan rate is an indication that the $Cu(NH_3)_4^{+}$ and $Cu(NH_3)_4SO_4$ reduction to Cu proceeds through a diffusion-controlled process.

Received: 09 Janurary 2020
Revised: 09 February 2020
Accepted: 08 July 2020

DOI: 10.3329/bjsir.v55i3.49397

Keywords: Copper ore; Leaching; Cyclic voltammetry; Electrodeposition; Electro winning

Introduction

The importance of copper for applications in electronic devices such as electronic circuit board, transmission of electricity and electroplating purposes has continued to rise due to industrial expansion to meet with the increasing demand. The metal is usually obtained by electro deposition from aqueous solutions after leaching (Popescu *et al.,* 2013). Copper electro deposition from alkaline solutions has been widely applied for industrial coating of materials and electro winning of the metal (Alam *et al.,* 2007; Oishi *et al.,* 2007; Popescu *et al.,* 2013). One of the notable ways of understanding the behaviour of copper and deposition processes in solution is by investigating its electrochemical reactions. The approach has been shown to be of great advantage over other methods with regards to electrolytic reduction and kinetics of the metal deposition from solutions (Majidi *et al.,* 2009; Ballesteros *et al.,* 2011). Cyclic Voltammetry (CV) analytical technique is often usedfor the elucidation of the reactions occurring during the electrodeposition processes due to its relative ease of operation and high information content (Nila and González, 1996a; Lanez and Neghmouche, 2013). In addition, the technique is mostly preferred because of its simplicity, compatibility, and good control over vacuum-based deposition processes (Grujicic and Pesis, 2005; Majidi *et al.,* 2009; Sophia and Muralidharan, 2015).

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Electrodeposition of copper can be carried out typically in a complexing agent such as Ethylenediamine tetra acetic acid (EDTA) and ammonia (NH_3) (Koyama *et al.,* 2006; Viswanatha and George, 2011). Factors influencing the rate of copper depositionsuch as pH, stability of complexes and concentration of ions from ammonium chloride complexes have been investigated (Nila and González, 1996a). Ammonium salts and other complex forming reagents are widely used for leaching copper from high gangue complex ores (Ekmekyapar *et al.,* 2012; Ochromowicz *et al.,* 2014). The recovery process of the metal from the solution containing the metal complex can be carried out by adsorption, electro winning or solvent extraction (Nila and González, 1996a and 1996b; Awe and Sandström, 2010; Liu *et al.,* 2012). The electrodeposition of copper from leachate obtained from the leaching of the ore in ammonium chloride lixiviant has scarcely been investigated.

In this present study, a Near Infrared (NIR) pre-concentrated copper ore was leached with ammonium chloride solution according to the method described by Baba and Adekola (2010), Baba *et al.* (2013) and Amos *et al.* (2019). The leachate obtained from the leaching of the ores containing copper ammonia complexes was subjected to cyclic voltammetry analysis to understand the electrochemical behaviour of copper from the complexes. The electrodeposition process and mechanism controlling the processwas also investigated in order to generate data for developing an efficient copper recovery process. The influence of scan rates on the reduction of copper was also observed. A synthetic electrolyte containing copper ammonium sulphate was also examined and results of the electrodeposition behaviour of copper in the synthetic electrolyte compared with that of the ore leachate.

It should be noted that before the leaching and electrochemical studies was carried out on the leachate, the influence of interfering metals (Mn, Zn, Co and Ni) was minimised by first pre-concentrating the ore after the method described by Iyakwari *et al.* (2013). Subsequently, the high grade copper ores was selectively leached (Amos *et al.,* 2019).

Materials and methods

Chemicals were purchased from VWR international Ltd, UK. All solutions used were freshly prepared from analytical-reagent grade chemicals without further purification. Leachate was obtained after leaching of copper oxide ore in 1 M NH_4Cl for 2 h. Solution pH of leachate was 8.5, the synthetic solution of copper ammonium sulphate ($Cu(NH_3)_4SO_4$ was prepared using $CuSO_4.2H_2O$ and ammonia and pH adjusted to 9 unless otherwise stated. The flow diagram for the strategy of ore pre-concentration, leaching and electrochemical analysis is presented in Fig. 1.

Leaching operation

Prior to leaching the ore was crushed, ground and sieved to the desired particle size fraction of -63/+45 μm amenable particle size for effective chemical leaching. The leaching experiment was performed using 250 ml of freshly prepared aqueous solution of NH_4Cl of known molarity in a 500 ml reactor with a four-neck split flask in a thermostatically heating mantle with a temperature control unit. The reactor is designed with an overhead mechanical stirrer for agitation set at 300 rpm for 2 h and a rubber stopper for sampling of leachate. The desired temperature for leaching is obtained by adjusting the thermostatically-controlled electric heating mantle. 5 ml of leachate solution for electrochemical analysis was withdrawn and filtered using filter paper (Whatman No.540). The filtrate was then advanced for analyses as described in Fig.1.

Cyclic voltammetry

The cyclic voltammetry measurements were conducted using external bi-potentiostat from CHI Electrochemical Analyser Model 660D. It contains a 100 ml polarographic cell fitted with a three-electrode configuration. The working electrode (WE) consisted of 0.08 cm^2 and 0.5 cm^2coiled platinum wire, the counter electrode (CE) was made of platinum sheet, and the reference electrode (RE) for cyclic voltammetric studies was leak-free Ag/AgCl in a 3 M KCl solution. All potentials are quoted with reference to the standard hydrogen electrode (= 0.222V). Potentiodynamic experiments were conducted across a range of cathodic potentials from 0.8 V to -1.0 V for selected scan rates (20, 50, 100, and 200 mV/s). All the experiments were carried out at room temperature ($T = 25\ ^\circ C$).

Results and discussion

Electrochemical studies

Fig.2 show the cyclic voltammogram obtained from solution of copper ore leachate. The potential scan rates of

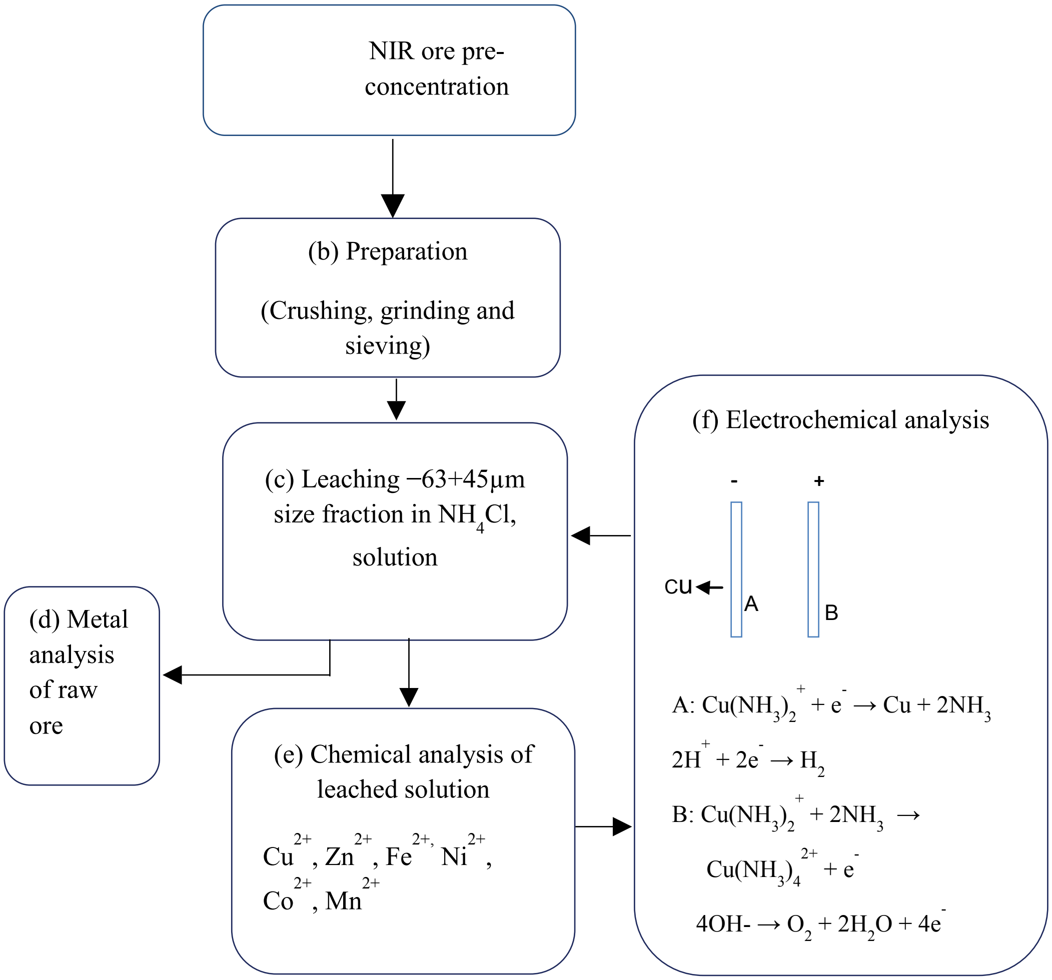

Fig. 1. Flow diagram of the strategy for pre-concentration, leaching and electrochemical analysis

20, 50, 100, and 200 mV/s in the cathodic and anodic directions were carried out. The starting potential was froma positive potential of 0.8V to a negative potential of -1.0 V. The excursion from the positive to negative potential reveals two reduction peaks (A and B) at cathodic potentials between -0.10 and -0.23V. Similarly, in the reverse scan (anodic) direction, two peaks (C and D) were also observed at anodic potentials of around -0.10 and 0.39V. The two cathodic peaks A and B are a result of the divalent copper ammonia complex undergoing reduction to the monovalent copper complex (peak A) and subsequently to copper metal (peak B). The peak depth increase with increasing scan rate in B suggests the electrodeposition of copper on the platinum electrode takes place at a negative potential between -0.10 and -0.23V. The two- step reduction process occurring in peaks A and B is expressed in Equations 1 and 2, respectively.

$$Cu(NH_3)_4^{2+} + e^- \quad Cu(NH_3)_2^{+} + 2NH_3 \qquad (1)$$

$$Cu(NH_3)_2^{+} + e^- + 2NH_3 \qquad (2)$$

The $Cu(NH_3)_4^{2+}/Cu(NH_3)_2^{+}$and $Cu(NH_3)_2^{+}/Cu$ corresponds to Cu(II)/Cu(I) and Cu(I)/Cu(0) redox couples and their potential are depended on the nature of electrode/ electrolyte interface. Also, the cathodic peak potential varies with sweep rates (Fig.2). The observation (Equations 1 and 2) are consistent and similar with that by Brown and Wilmott (1985) and Majidi *et al.* (2009). This suggests that it involves a fast electron transfer process.

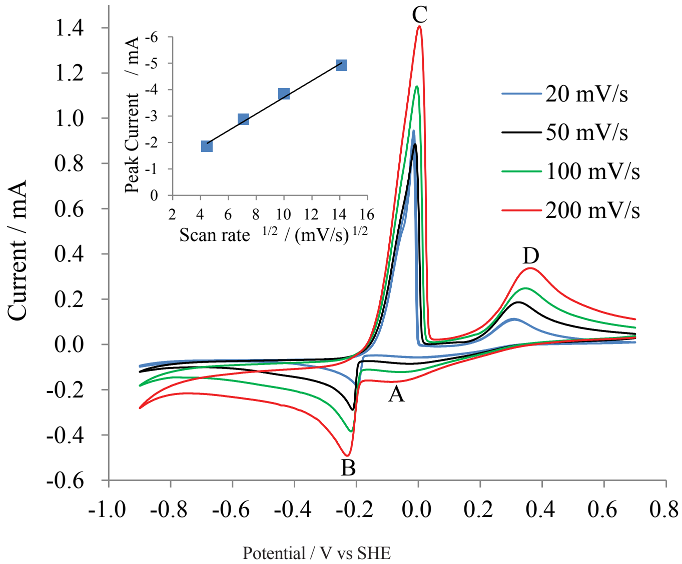

Fig. 2. Cyclic voltammograms of ore leachate at different scan rates. Inset: Cathodic peak current versus square root of scan rate

During the reverse scan, electrochemical regeneration of $Cu(NH_3)_4^{2+}$ complex takes place by the dissolution of copper and the formation of monovalent copper ammonia complex in the presence of the complexing agent. Two peaks (C and D) wereobserved which have different width to the forward scan peaks (A and B). The width of peak C suggests that the electro active species deposited (shown by peak B) undergoes a stepwise dissolution in the anodic or reverse scan direction. Equation 3 shows the oxidative dissolution of copper in the presence of ammonia (peak C):

$$Cu + 2NH_3Cu(NH_3)_2^{+} + e^{-} \qquad (3)$$

The monovalent copper (I) ammonia complex is unstable and undergoes oxidation into the stable copper (II) ammonia complex. This oxidation, which is facilitated by oxygen in the solution, is indicated by the sharp anodic stripping peak D and by the high value of peak current. The equations for this reaction are presented in Equations 4 and 5 (peak D):

$$Cu + Cu(NH_3)_4^{2+} 2Cu(NH_3)_2^{+} \qquad (4)$$

$$Cu(NH_3)_2^{+} + 2NH_3 + ½O_2 + H_2O + e^{-} \rightarrow Cu(NH_3)_4^{2+} + 2OH^{-} \qquad (5)$$

The increase in the peak C height as a function of scan rate reflects that the reduction of the copper (I) ammonia complex occurred as a reversible process while the peak D corresponds to a single electron transfer process. The clear difference in the peak areas of anodic peak C and cathodic peak A suggest that the reaction is partially reversible. Thus the closeness in the current of peaks (A and D) attributed to the reversible reactions suggest that the redox couple Cu(II)/Cu(I) is a reversible system. The electrochemical reaction in peaks C and D is an indication of the reversibility of electrodeposition and dissolution of copper on platinum electrode. This is in line with the observation by Popescu *et al.* (2013).

The difference in the number of ammonium ligands in the two peaks A and B(Equations 1 and 2) corresponding to [Cu(II)/Cu(I)] redox couple indicates that the overall reaction involves the deco ordination of ligands. Shumilov *et al.* (1983) and Brown and Wilmott (1985) showed that the reduction occurs on the triammonia complex ($Cu(NH_3)_3^{+}$) . This observation is in linewith the one reported by Darchen *et al.* (1997). Aravinda *et al.* (2000) reported that the electronation of divalent copper (II) solutions containing complexing agents occur by a stepwise process with ammonia and a number of ammine and nitrogen-containing heterocyclic compounds given rise to electronation of the copper complexes. The two-step reductions of copper complexes were observed

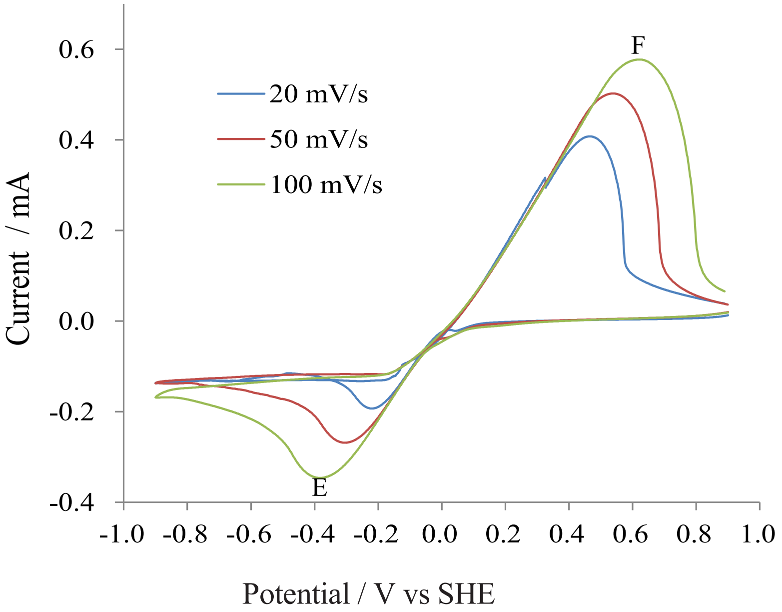

Fig. 3. Cyclic voltammograms of synthetic $Cu(NH_3)_4SO_4$ electrolyte at different scan rates. Inset: Cathodic peak current versus square root of scan rate

by potentiometry (Darchen *et al.,* 1997; Giannopoulou *et al.,* 2009).

Fig.3 shows the cyclic voltammogram of syntheticcopper ammonium sulphate solution. A single pair of redox peak is observed for the complex, corresponding to Cu/Cu^{2+}redoxcouple as indicated by the cathodic and anodic peaks (E and F). The appearance of the two peaks instead of the four in Fig.3is an indication that the electrodeposition behaviour is different. Anodic Peak F signifies the dissolution of copper electrodeposited during the forward potential scan. Peak E suggests that the electro active copper (II) is reduced, undergoing electron transfer predominantly from the copper ammonia complex. The unique electrochemical reactions observed for the $Cu(NH_3)_4SO_4$ solution is an indication of a quasi-reversible deposition/dissolution behaviour of Cu^+ion occurring in unison. The electrochemical behaviour observed is similar to that by Popescu *et al.* (2013). Thus the reduction of Cu^{2+} to Cu metal during the cathodic scan and its oxidation in the anodic scan is an indication of the reversibility of the reaction, as shown in Equations 6 (peak E) and 7 (peak F):

$$Cu(NH_3)_4^{2+} + e^- \quad Cu(NH_3)_2^+ \quad + 2NH_3 \qquad (6)$$

$$Cu(NH_3)_4^+ + e^- \quad Cu + 4NH_3 \qquad (7)$$

Equations 6 and 7 suggest that $Cu(NH_3)_4^+$ is an intermediate species during the reduction of $Cu(NH_3)_4^{2+}$, which proceeds through a ligand-coupled electron transfer reaction and involves the conversion of the ammonium ion. Pecequilo and Panossian (2010) also used cyclic voltammetry technique to understand the electrodeposition mechanism of copper from strike alkaline bath and showed that Cu^{2+}reduction to Cu occurs via a direct reduction without formation of Cu^+ inter mediate. Majidi *et al.* (2009) and Meng and Bard (2015) indicated that the reduction of copper can occur by either ion transfer controlled regime or via ligand-coupled electron transfer reaction. This is consistent with the observations in Fig.3.

Electrochemical kinetics

Figs. 2 and 3 can be used to infer the electrodeposition kinetics. The cyclicvoltammograms obtained from the various scans show that the behaviour of ore leachate and synthetic solution are distinct. The cathodic peak potential (B), for the reaction given by Equation 2,is -0.20, -0.21, -0.22 and -0.23 V at the scan rate of 20, 50, 100 and 200 mV/s, respectively (Fig. 2). The differences between the cathodic and anodic potentials are 0.19, 0.20, 0.21 and 0.23 V, only increased slightly with the scan rate. The gradual shift of oxidation/reduction peaks with scan rate is evidence of reversibility of electrochemical process taken place. As shown in the inset of Fig.2, the cathodic peak current exhibits a linear relationship with the square root of the scan rate, suggesting that reduction of Cu^{2+} to Cu is mainly under diffusion control. The observed electrochemical behaviour is similar with that by Grujecic and Pesic (20015) and Popescu *et al.* (2013). The higher peak currents and smaller peak width provides further evidence of a highly reversible reaction. The cathodic peak (E) for the synthetic solution of $Cu(NH_3)_4SO_{4,}$ which correspond to Cu^{2+}/Cu redox couple, appear at -0.22, -0.30 and -0.38 V at the scan rate of 20, 50 and 100 mV/s respectively, (Fig.3). The difference between the cathodic and anodic peak potentials increased with the scan rate, which is characteristic of slow electron transfer kinetic. Therefore, comparison between the two experiments shows that the Cu^{2+}/Cu redox couple in the synthetic solution is a sluggish electron transfer process due to the large separation between the cathodic and anodic peak while the $Cu/[Cu(NH_3)_2]^+$ redox reaction in the ore leachate is more reversible, with fast electron transfer. While only one pair of redox peaks are observed for $Cu(NH_3)_4SO_4$ solution, this suggests two separate one electron transfer reactions attributed to simultaneous cathodic deposition of ionic Cu^{2+} to copper and anodic dissolution of copper to Cu^{2+}. The inset of Fig.2 shows that the relationship between the peak current and the square root of the scan rate is linear; indicating that reduction of Cu^{2+} to Cu (0) is also a diffusion-controlled process. With two pairs of redox peaks observed for the ore leachate, this indicates that the reduction of $[Cu(NH_3)_2]^+$ to Cuhas a higher rate constant compared to the reduction of $Cu^{2+}$to Cu. As a result, when the peak current C increases peak A, which corresponds to the reduction of Cu(II) to Cu(I), also increasesconsistently.

The cyclic voltammograms curves provide insight into the electrochemical behaviour and electrodeposition of copper from ore leachate solution and $Cu(NH_3)_4SO_4$synthetic solution. The increase in copper deposition Cu (I) Cu (0) increases as a response to increase in scan rate. It was also observed that the increase in scan rates led to remarkable increase of both cathodic and anodic peaks in response to increase in current density. The observed behaviour depends on potential sweep rates, as presented in the Figs. 2 and 3. From the voltammetric scans, it is clear that the copper reduction is a stepwise process involving two steps, with the electronation of the Cu^{2+} in the solution containing the complexing agent. The electrodeposition is an equilibrium process whereby the free Cu^{2+}concentration is in equilibrium with Cu^+ concentration. Under transient polarisation conditions, the deposition of copper involves the formation of monovalent copper and the ammonia ligand. The reversibility of the Cu(II)/Cu(I) reactions established with the triammonia copper complex (Equation 8) as the intermediate specie in the redox reaction (Brown and Wilmott, 1985).

$$Cu(NH_3)_3^{2+} + e^- \quad Cu(NH_3)_3^{+} \qquad (8)$$

It is observed that the electrodeposition of copper is similar to the conventional electrowinning process of the metal which is known to proceed according to a two-step process. The kinetics of the electrochemical reactions indicated that copper deposition in both the solutions were under diffusion-controlled mass transfer process.

Conclusion

Electrochemical studies of two solutions of copper complexes were successfully investigated. The deposition behaviour of copper from solution of ore leachate was studied with cyclic voltammetry and compared with a synthetic electrolyte solution of $Cu(NH_3)SO_4$. The influence of scan rate and potential on the two solutions was investigated. It was found that the deposition of copper metal from the leachate solution is a two-step process while that of the copper ammonium sulphate consist of two separate one-electron transfer processes. The electrodeposition increases with increasing scan rates. The transition from the divalent state to the monovalent state is a reversible process, with electron transfer being a fast process. This suggests that the reactions are diffusion controlled.

Acknowledgment

The Nigerian Government, through the academic staff support scheme of the Tertiary Education Trust Fund of Nigeria (TET fund) and the Management of Nasarawa State University, Keffi, are acknowledged for providing financial support for this research

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