

Cyclic Voltammetry Study to Investigate the Effect of Various Parameters on Cadmium Electrodeposition in Choline Chloride Based Ionic Liquid

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Abstract: Cyclic voltammetry (CV) is a powerful and popular electrochemical technique commonly employed to investigate the reduction and oxidation processes of molecular species. It has been used to investigate the interaction behavior of cadmium from a solution containing cadmium chloride ($\text{CdCl}_2 \cdot \text{H}_2\text{O}$) in an ethylene glycol (EG)-choline chloride (ChCl) based ionic liquid has been carried out onto a copper cathode by constant current and constant potential methods at room temperature. The influences of various experimental conditions and some parameters on cyclic voltammetry have been investigated using a Potentiostat/Galvanostat. It was observed that deposition occurs in $\text{ChCl}:2\text{EG}:0.1\text{M}\text{CdCl}_2 \cdot \text{H}_2\text{O}$ ionic liquid via an instantaneous nucleation mechanism in the very initial stage of the crystal growth. It is shown that smooth and good adhering bright metallic coloured cadmium has been electrodeposited from this ionic liquid (ethaline) containing $\text{CdCl}_2 \cdot \text{H}_2\text{O}$ at room temperature in presence of $0.10 \text{ g}\cdot\text{L}^{-1}$ polyethylene glycol and $40 \text{ mL}\cdot\text{L}^{-1}$ formic acid mixture and $0.05 \text{ mol}\cdot\text{L}^{-1}$ P_2O_5 as additives. The cadmium electrodeposit obtained at the applied deposition potentials up to -0.95 V and current densities up to $-20.0 \text{ A}\cdot\text{m}^{-2}$ are very smooth, good adhering and of uniform size. Cadmium electrodeposits cannot be obtained from urea/glycerol based ionic liquid (reline/glyceline) at any temperature and even on the addition of additives.

Keywords: *Cyclic Voltammetry, Electrodeposition, Ethaline, Reline, Nucleation.*

Introduction: The most used method for gathering qualitative data regarding electrochemical reactions is cyclic voltammetry. It provides quick localization of the electroactive species' redox potentials. By relating kinetic and experimental parameters, it is possible to create diagnostic criteria that can be used to characterize unknown systems by examining changes in peak current, half-peak potentials, or the ratio of anodic to cathodic peak currents as a function of the rate of voltage scan. The effect of a chemical reaction on the voltametric wave will depend on its rate, as compared with the time required to perform the experiment. The ratio of the rate constant to the rate of voltage scan appears in the kinetic parameter.

Cadmium is a naturally occurring metal situated in the Periodic Table of the elements between zinc and mercury. It is soft, malleable, ductile, bluish-white divalent metal. It is similar in many respects to zinc [1]. Unlike most other metals, cadmium is resistant to corrosion and is used as a protective coating on other metals. As a bulk metal, cadmium is insoluble in water and is not flammable; however, in its powdered form it may burn and release toxic fumes [2]. Commercially, cadmium is used in television screens, lasers, batteries, paint pigments, cosmetics and in galvanizing steel, as barrier in nuclear fission, and has been used with zinc to weld seals in lead water pipes prior to the 1960s.

Cadmium coatings have many uses because they have good corrosion resistance in saline environment, have low friction coefficient and make fair electrical contact. Cadmium coatings are used mostly in components for the electrical, electronic, communications, automotive, aeronautic and locks industries. Cadmium electroplating is used in the aircraft industry to reduce corrosion of steel components [3]. It has been proven that cadmium forms a very satisfactory plate on steel, which has to withstand severe corrosion, especially the corrosion of seawater and spray. The metal is now successfully plated electrolytically from cyanide solutions, but wherever work is carried on with cyanide, there is great danger of poisoning to the workers. With these dangers in mind, it can be readily seen that if a satisfactory substitute could be found for the cyanide electrolyte in cadmium plating, it would be of great value. Cadmium sulfate solutions are now widely used in the electro-winning of cadmium, but so far have a very limited application in the electroplating industries. A. Dolati et al. [4] and W. T. Tan [5] have reported the electrodeposition of cadmium from sulfate and chloride solutions, respectively. The hydrogen release generated from aqueous solutions always results in the hydrogen embrittlement of cadmium deposits, low current efficiency, and high energy consumption. Hydrogen embrittlement on cadmium coatings is greater in acid plating baths. This problem is overcome by using room temperature ionic liquid (RTIL) as an electrolytic bath for the deposition of cadmium.

RTILs composed of entirely of organic cations and organic/inorganic anions are a viable alternative to the aqueous solution due to negligible vapour pressure at elevated temperature, good ionic conductivity, good thermal stability, and wide electrochemical window [6-10]. In recent years they have been used as electrolyte for various electrochemical applications including

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electrodeposition with high current efficiency [8-13]. Mixtures of quaternary ammonium salts, in particular choline chloride, with hydrogen bond donors like amides, alcohols and carboxylic acids which are known as deep eutectic solvents (DES) [14, 15] and can be an alternative ionic liquid (ILs). These ILs have the same advantages as RTILs but they are cheaper, air and water stable [14, 15]. These ILs have been used for the deposition of a range of metal coatings including Zn [16], Cr [17], Sn [18], Cu [19, 20], Ni [21, 22] and Ag [23, 24] at high current efficiency. The Effect of water presence on choline chloride-2urea IL and coating platings from the hydrated IL has been reported by Cuiling Du *et al.* [25].

The electrochemical behavior of cadmium ions has been studied in ionic liquid at different conditions to determine the deposition conditions of metallic cadmium; the authors reported that both the ionic liquid and the deposition conditions strongly influence the quality and properties of the deposited layers.

This paper presents an electrochemical study of cadmium ions behaviour in an ionic liquid named ethaline in presence of surfactants at room temperature to 60°C temperature range on platinum electrode using cyclic voltammetric technique.

EXPERIMENTAL DETAILS

CHEMICALS: Choline chloride ($\text{HOC}_2\text{H}_4\text{N}^+(\text{CH}_3)_3\text{Cl}$) ChCl (Aldrich 99%) was, when necessary, recrystallized from absolute ethanol, filtered and dried under vacuum. Ethylene glycol (EG) ($\text{HOCH}_2\text{CH}_2\text{OH}$) (Aldrich > 99%) was used as received. Cadmium chloride monohydrate ($\text{CdCl}_2\cdot\text{H}_2\text{O}$) (Loba Chemie 98%) was used as obtained. Solution was generally made up to a metal halide concentration in the ranging from 0.025 to 0.2 $\text{mol}\cdot\text{L}^{-1}$.

The additives di-phosphorus pentoxide (P_2O_5) (Merck >97%) and acetonitrile (CH_3CN) (Merck >99.9%) were used as received. Generally, the additives were added to the plating bath to a concentration of 0.10 $\text{g}\cdot\text{L}^{-1}$ polyethylene glycol and 40 $\text{mL}\cdot\text{L}^{-1}$ formic acid mixture or 0.05 $\text{mol}\cdot\text{L}^{-1}$ (P_2O_5) or 40 $\text{mL}\cdot\text{L}^{-1}$ acetonitrile. All other chemicals were used as received.

PREPARATION OF IONIC LIQUID: The eutectic mixture was formed by stirring the two components together, in the stated proportions, at 75°C until a homogeneous, colourless liquid formed. The molar ratio for the eutectic compositions was found to be 1:2 for choline chloride (ChCl) with ethylene glycol (EG). The ionic liquid, once formulated, was kept in a thermostatic oven at 30°C prior to use.

ELECTROCHEMICAL MEASUREMENTS: Electrochemical investigations including potential step chronoamperometry, chronopotentiometry and cyclic voltammetry were carried out using a Hokudo Denko HAB-151 Potentiostat/Galvanostat, Tokyo, Japan, equipped with a potential sweeper. Data were recorded in a computer through data acquisition system (USA) using WinDaq software. A three-electrode system consisting of a platinum ($50 \times 5 \times 0.1 \text{ mm}^3$) working electrode, a platinum ($50 \times 5 \times 0.1 \text{ mm}^3$) counter electrode and a silver wire quasi-reference electrode were used in all electrochemical studies. The working and counter electrodes were cleaned electrochemically in 1.5 $\text{mol}\cdot\text{L}^{-1}$ H_2SO_4 –1.5 $\text{mol}\cdot\text{L}^{-1}$ H_3PO_4 mixtures, then rinsed with deionised water, and finally dried with acetone prior to use in all measurements. All voltammograms were obtained at 30°C with various scan rates ranging from 5 to 100 $\text{mV}\cdot\text{s}^{-1}$. A silver wire (immersed in ethaline) quasi-reference electrode was used in all electrochemical experiments which have been shown to have stable reference potential in chloride-based eutectics. All potentials in this work are quoted with respect to this $\text{Ag} | 1:2 \text{ ChCl-EG}$ reference electrode which will be written as $\text{Ag} | \text{Ag(I)}$ (note the reference potential will change slightly with melts of different compositions). The overall instrumental cell set-up for the cyclic voltammetry experiment is shown in figure 1.



Fig. 1 The instrumental set-up of the three electrodes cell connected with the potentiostat/galvanostat via transducer with winDaq software installed computer system.

Results and Discussion

Cyclic voltammetry of Cd(II) in ChCl:2EG (ethaline) IL: The cyclic voltammogram recorded on a platinum electrode in 1:2:0.10 (mole ratio) ChCl:EG:CdCl₂·H₂O IL at 30°C with a scan rate of 10 $\text{mV}\cdot\text{s}^{-1}$ is shown in Fig. 2. The rest potential is +0.45 V. The scan towards negative direction consists of first reduction wave C_1 with the current starting to increase at -0.70 V.

Additional reduction wave C_2 is observed with the current again starting to increase at -1.12 V. The reverse scan consists of first (P_{a1}) and second (P_{a2}) stripping peaks at -0.54 V and -0.19 V, respectively. Additional oxidation wave is observed with the current again starting to increase at +1.15 V.

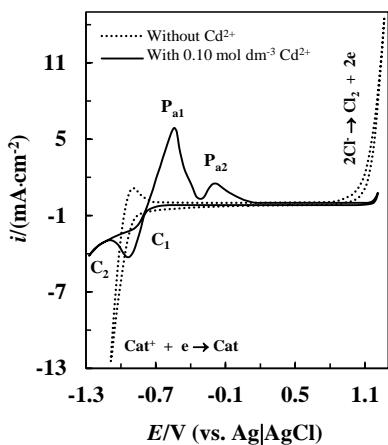


Fig. 2 Cyclic voltammogram recorded on a platinum electrode in ChCl:2EG IL containing $0.10 \text{ mol}\cdot\text{L}^{-1} \text{ CdCl}_2\cdot\text{H}_2\text{O}$ at 30°C with a scan rate of $10 \text{ mV}\cdot\text{s}^{-1}$.

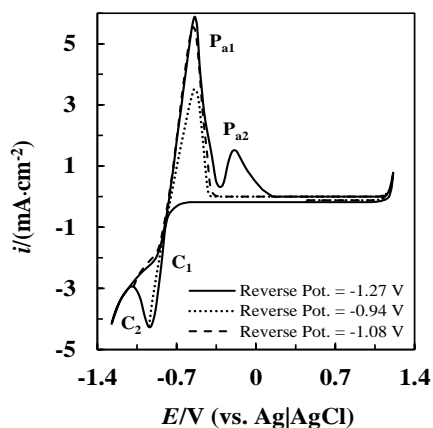


Fig. 3 Effect of sweeping potential on the cyclic voltammogram recorded on a platinum electrode in ChCl:2EG IL containing $0.10 \text{ mol}\cdot\text{L}^{-1} \text{ CdCl}_2\cdot\text{H}_2\text{O}$ at 30°C with a scan rate of $10 \text{ mV}\cdot\text{s}^{-1}$.

Compared with the voltammogram obtained in the absence of $\text{CdCl}_2\cdot\text{H}_2\text{O}$, dotted curve in Fig. 2, the reduction wave appeared at -0.72 V in ethaline corresponds to the reduction of cationic species (Cat^+) into this IL, while the oxidation wave appeared at +1.2 V in ethaline corresponds to the oxidation of chloride ions (anions) to molecular/gaseous chlorine [22]. Fig. 3 shows the effect of cathodic sweeping potentials on the cyclic voltammogram recorded on a platinum electrode in 1:2:0.10 (mole ratio) ChCl:EG: $\text{CdCl}_2\cdot\text{H}_2\text{O}$ IL at 30°C with a scan rate of $10 \text{ mV}\cdot\text{s}^{-1}$. It is readily seen from the voltammogram that the first reduction wave (C_1) corresponds to the first oxidation peak (P_{a1}).

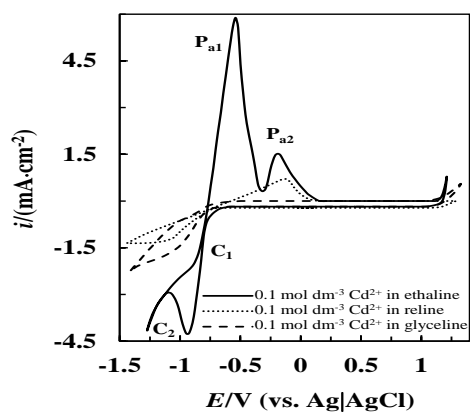


Fig. 4 Effect of electrolytes (ILs) on the cyclic voltammogram recorded on a platinum electrode at 30°C with a scan rate of $10 \text{ mV}\cdot\text{s}^{-1}$.

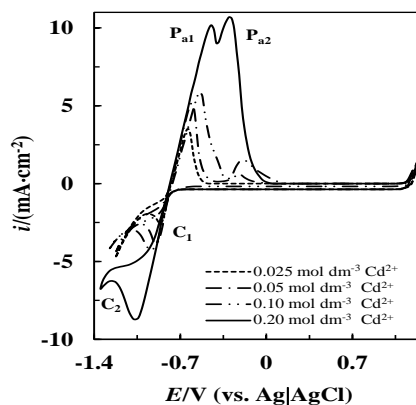


Fig. 5 Effect of $\text{CdCl}_2\cdot\text{H}_2\text{O}$ concentrations on the cyclic voltammogram recorded on a platinum electrode in ChCl:2EG IL at 30°C with a scan rate of $10 \text{ mV}\cdot\text{s}^{-1}$.

From XRD analysis, pure cadmium has been detected in the deposit obtained at a deposition potential of -0.92 V (from the first reduction wave C_1) by constant potential method. Therefore, the increase of the cathodic current in the first reduction wave (C_1) is obviously associated with the reduction of cadmium ion to metallic state according to the following reaction:



When the potential sweep is made progressively more negative than -1.12 V, the second stripping peak P_{a2} begins to develop at about -0.19 V. This reduction wave (C_2) is attributed to the reduction of cationic species of IL as compared with dotted curve in Fig. 2. However, cadmium is also deposited in this potential range. This strongly suggests the presence of two energetically different phases/morphologies of cadmium deposit. The two different morphologies must result from different growth mechanisms. One possibility could be the initial growth of a large number of nuclei on the surface where some of which stop growing giving a material with a different morphology. Alternatively, some aspect of the interfacial layer structure could

neutralise the surface energy of the growing particle. Similar morphology has been reported by Abbott *et al.* [26] for the deposition of aluminium from chloroaluminate based ionic liquid and Ali *et al.* [24] for the deposition of silver from reline.

Figure 4 shows the effect of electrolytes (ILs) on the cyclic voltammograms recorded on a platinum electrode at 30° C. The dotted and dashed curves show the cyclic voltammograms of 0.10 mol·L⁻¹ Cd²⁺ in reline (ChCl:2urea) and glyceline (ChCl:2glycerol), respectively. It is readily seen from the voltammograms that a sharp stripping and reduction peaks are obtained in solid curve indicating the deposition of cadmium is possible in ethaline. However, very small reduction and stripping peaks are obtained in reline and glyceline. Pure Cd deposit could not obtain from these two electrolytes (reline and glyceline) by constant potential or current methods. Therefore, ethylene glycol-based IL (ethaline) has been selected for the deposition of cadmium.

Figure 5 shows the effect of CdCl₂·H₂O concentrations on the cyclic voltammograms recorded on a platinum electrode in ChCl:2EG IL at 30° C with a scan rate of 10 mV·s⁻¹. It is readily seen from these voltammograms that the magnitude of current density in the reduction wave (C₁), which is attributed to the reduction of Cd(II) to Cd(0), increases with the increase of the CdCl₂·H₂O concentrations added into the IL. The same phenomenon is also observed with the stripping peak (P_{al}), which is also show the increase of the current density with the increase of the CdCl₂·H₂O concentrations added into the IL. The increases in the magnitudes of Cd(II) reduction current densities with the increase of CdCl₂·H₂O concentrations added into the IL is due to increased mass transport, which would act to promote the diffusion of cadmium ions to the electrode surface, encouraging bulk growth. Similar result has been reported in the previous studies for the deposition of Cu [22] and Ag [24] in this IL.

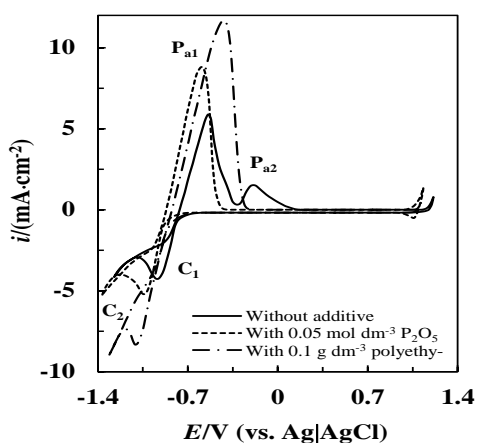


Fig. 6: Effect additives on the cyclic voltammograms recorded on a platinum electrode in ChCl:2EG IL containing 0.1 mol·L⁻¹ CdCl₂·H₂O at 30° C with a scan rate of 10 mV·s⁻¹.

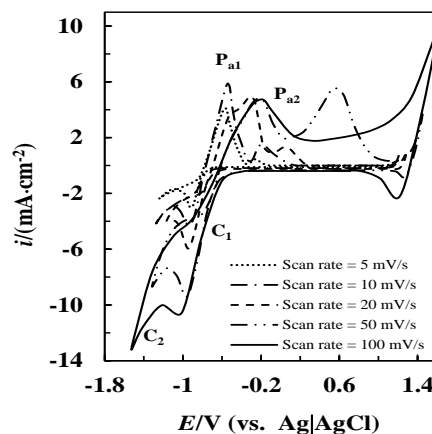


Fig. 7: Effect scan rate on the cyclic voltammogram recorded on a platinum electrode in ChCl:2EG IL containing 0.10 mol·L⁻¹ CdCl₂·H₂O at 30° C.

The effects of additives on the cyclic voltammogram recorded on a platinum electrode in ChCl:2EG:0.1CdCl₂·H₂O IL at 30° C with a scan rate of 10 mV·s⁻¹ are presented in Fig. 6. It is clear from Fig. 6 that the presence of additives causes significant changes in the shape and position of the voltammograms.

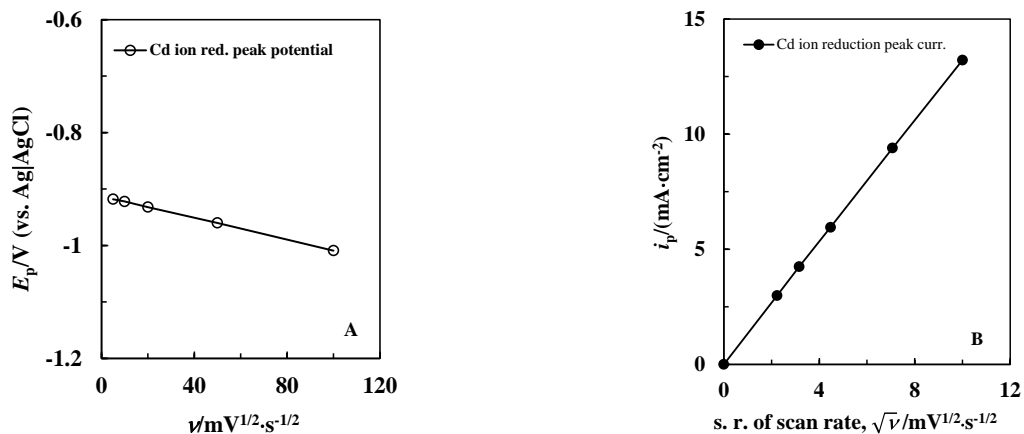


Fig. 8 Variation of cathodic peak current densities with the square root of scan rate for the reduction of cadmium ion in ethaline containing 0.10 mol·L⁻¹ CdCl₂·H₂O.

The addition of $0.10 \text{ g}\cdot\text{L}^{-1}$ polyethylene glycol & $40 \text{ mL}\cdot\text{L}^{-1}$ formic acid mixture, dot and dashed curve in Fig. 6, has the largest effect on the voltammetric response. It is interesting to note that the magnitude of the reduction and stripping current densities is significantly increased and the second stripping peak is disappeared upon the addition of additives into the IL. This is likely to simply be an effect of increased mass transport due to the addition of additives in IL. However, the onset of Cd reduction is shifted cathodically by 100 mV to $E = -0.80 \text{ V}$ upon addition of additives, suggesting that in this system the additives inhibit the initiation of Cd nucleation.

The effects of scan rates on the cyclic voltammograms recorded on a platinum electrode in $\text{ChCl}:2\text{EG}:0.1\text{CdCl}_2\cdot\text{H}_2\text{O}$ IL at 30°C are presented in Fig. 7. For a reversible system, peak potential E_p is independent of scan rate (ν), and peak current density (i_p) is proportional to the square root of scan rate ($\sqrt{\nu}$). Variations in the peak potentials with scan rate (E_p vs. ν) and peak current densities i_{pc} with square root of scan rate (i_p vs. $\sqrt{\nu}$) are shown in Fig. 8(A) and Fig. 8(B), respectively. It is readily seen from Fig. 8(A) that the peak potentials for the redox reaction of $\text{Cd(II)}/\text{Cd(0)}$ couple is almost independent of the low scan rates, indicating a reversible in nature. However, at high scan rates the peak potential is not independent of the scan rates because the peak is somewhat broad, so that the peak potential may be difficult to determine. According to Randles-Sevcik equation [27], the linear increases in the cathodic peak current densities with the square root of scan rate, as shown in Fig. 8B, indicate that the reduction process of cadmium ion in $\text{ChCl}:2\text{EG}$ based IL is controlled by diffusion.

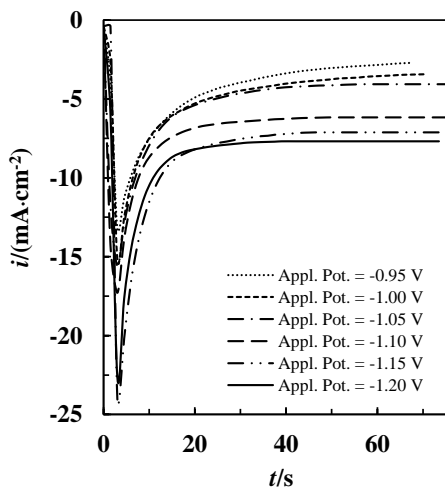


Fig. 9 Potentiostatic current-time transients for the deposition of cadmium from $\text{ChCl}:2\text{EG}:0.1\text{CdCl}_2\cdot\text{H}_2\text{O}$ IL at 30°C and different applied potential.

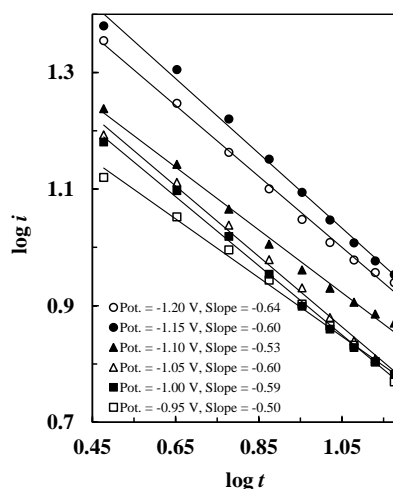


Fig. 10 Plots of $\log i$ vs. $\log t$ for rising part of the current-time transients in Fig. 9.

Fig. 9 shows the potentiostatic $i-t$ transients measured on a platinum electrode from 1:2:0.1 mole ratio of $\text{ChCl}:\text{EG}:\text{CdCl}_2\cdot\text{H}_2\text{O}$ IL at different applied potentials. The experimental details and discussion are similar to those in the previous study [28-30]. Figure 10 shows the $\log i$ versus $\log t$ plot for the rising part of $i-t$ transients in Fig. 9. The measured slopes at different applied potentials for the deposition of pure cadmium are between 0.5 and 0.64, which are quite close to 0.5. This suggest that the nucleation mechanism of cadmium deposition from 1:2:0.1 (mole ratio) $\text{ChCl}:\text{EG}:\text{CdCl}_2\cdot\text{H}_2\text{O}$ IL most probably proceeds instantaneously at the very initial stage of the crystal growth. The diffusion coefficient for the electrodeposition processes of cadmium has been estimated from Fig. 9 and 10. The average values of the diffusion coefficient are $2.453 \times 10^{-5} \text{ cm}^2\cdot\text{s}^{-1}$ and $1.655 \times 10^{-4} \text{ cm}^2\cdot\text{s}^{-1}$, respectively.

Conclusions: This research work reveals that choline chloride-based IL specially ethaline can be used as electrochemical solvents. This work shows that choline chloride-based IL can be used as electrochemical solvents. Smooth and good adhering bright metallic coloured cadmium has been electrodeposited onto copper cathode from ethaline containing $\text{CdCl}_2\cdot\text{H}_2\text{O}$ at room temperature in presence of additive. However, the deposits in absence of additive are not good adherence. The electrodeposition of cadmium from an $\text{ChCl}:\text{EG}:\text{CdCl}_2\cdot\text{H}_2\text{O}$ (1:2:0.1 mole ratio) IL occurs via an instantaneous nucleation mechanism in the very initial stage of the crystal growth. The cadmium electrodeposit obtained at the applied deposition potentials up to -0.95 V and applied deposition current densities up to $-20.0 \text{ A}\cdot\text{m}^{-2}$ are very smooth, good adhering and of uniform size. Cadmium electrodeposits cannot be obtained from urea/glycerol based IL (reline/glyceline) at any temperature and even on the addition of additives.

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