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RESEARCH ARTICLE

Copper electrodeposition using a novel Type IV deep eutectic solvent

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ABSTRACT

The research aimed to develop a new deep eutectic solvent (DES) composed of a mixture of lithium chloride and 1,3-propanediol, mixed at a 1:4 molar ratio, and used it for testing of electroplating of copper. Structural and electrochemical properties of copper electrolytes were determined with and without the use of the additives by using various electrochemical techniques, including cyclic voltammetric (CVs) measurements. Physical properties of the additive-free and additive-dispensed propanediol-based DES, such as electrical conductivity, were determined. The intent of this analysis was to determine how the additives present in the solvent altered the dissolving properties of the solvent, thus changing the electroplating process. The research further extends the knowledge of the newly introduced DES with respect to its impact on copper electrodeposition on nickel substrates. This evaluation was undertaken both with and without additives in order to assess their impact on the electroplating process. Important properties of the deposited copper, including thickness, surface shape, and surface roughness, were measured and compared. Finally, more sophisticated methods have been used to gain a deeper understanding of the copper deposits. The surface morphology of the deposits was analyzed by field emission-scanning electron microscopy (FESEM), and atomic force microscopy (AFM) was particularly useful in ascertaining the surface roughness. These methods enabled the observation and measurement of the microstructural variances of the copper deposits produced in different experimental setups.

Keywords: deep eutectic solvents; copper; electrodeposition; FESEM; AFM

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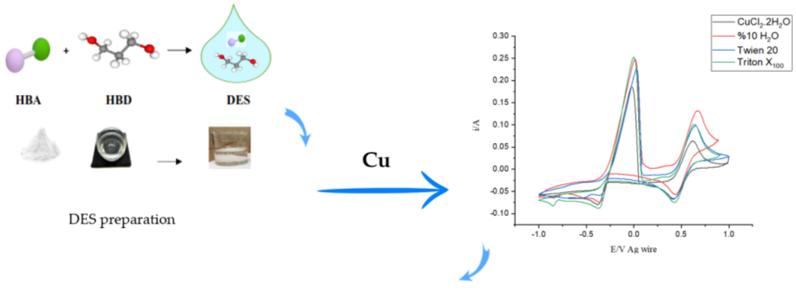
Graphical abstract



- ☐ Developed for Cu electrodeposition on Ni.
- ☐ H_2O, TritonX100, Tween20 improved Cu coating.
- ☐ TritonX100 and H_2O boosted Cu reduction.
- ☐ Additives (TritonX100, H_2O) reduced surface roughness and grain size.
- ☐ TritonX100 and H_2O resulted in well-adhered Cu films.
- ☐ Achieved hard, adherent Cu coating.

1. Introduction

Deep eutectic solvents (DESs), first presented by Abbott et al. in 2001, have emerged as intriguing and efficient substitutes for room temperature ionic liquids (IL) and organic solvents in a range of applications [1]. Studies comparing solutions that utilize ILs or DESs with those that rely on mixed solvents or traditional electrolyte solutions have indicated favorable outcomes, particularly in instances involving (DESs + H_2O) s.[2-4] and (IL + H_2O)[5-8]. Deep eutectic solvents (DESs) were first introduced through various studies that revealed their differences from ionic liquid systems. These solvents are produced by combining at least two starting substances and heating them, resulting in hydrogen bonding between a hydrogen bond donor[9,10] (HBD, such as alcohols or carboxylic acids) and a hydrogen bond acceptor (HBA, like a quaternary ammonium salt)[11]. The melting points of these DESs are much lower than those of their individual components, and they typically exist as liquids at room temperature[12]. As innovative and eco-friendly solvents, DESs have opened up new possibilities in sustainable practices[11]. Recently, deep eutectic solvents have emerged as a subset of modern solvents that share characteristics with ionic liquids. The terminology of deep eutectic solvents and ionic liquids has often been used interchangeably in various studies[8,3,14]. Essentially, deep eutectic solvents are ionic liquids that utilize a hydrogen bond donor as a complexing agent, and they can be described by an equilibrium equation:

Cation + anion + complexing agent ➞ cation + complex anion

or

Cation + anion + complex agent ➞ complex cation + anion[15]

DESs have gained popularity due, in general, to the versatility of its components and their biodegradability, high recycling rate, less waste disposed of during preparation[16]. DESs and ILs are similar in physical properties, and there are also differences between them, for instance, according to their molecular structures[7,10,17]. Ionic interactions govern the behavior of IL, which is mainly made up of an anion and a cation[16]. Additionally, DESs are more advantageous than ILs in a number of ways, including ease of preparation, low cost, greenness[11], suitability for large-scale industrial manufacturing[7,10,16], and non-volatility, low toxicity[18]. Industry applications for DESs include electropolishing, polymer preparation, electroplating, electrodeposition, etc.[19]. Therefore, electroplating in the industrial world (especially copper electroplating) is widely common to create conductive tracks in paths, circuit boards, decorative coatings, and sublayers[20]. Copper (Cu) is often regarded as the fundamental element in clean energy technology[21]. Copper was selected due to its well-documented deposition, and it’s, from a chemical standpoint, simple in formulating for the galvanic bath[22]. Vasiljevic has more recently published on the impact of anion adsorption on copper deposition[23]. The application of low halide concentrations had a significant impact on the copper film's shape; however this behavior was most likely caused by the partial production of CuX precipitates on the electrode surface, which altered the reaction pathway[22]. Many authors' recent studies have documented the impacts of different (organic, inorganic) additives on the baths of different metals (especially Cu) that are electrodeposited from aqueous and anhydrous electrolytes in order to that improve the deposit's composition, brightness, roughness, adhesion, electrochemical stability, and grain size[24]. Many researchers have investigated the effect of electrode placement additives on copper from aqueous solutions. The role played by additions in the electrodeposition from copper Cu of eutectic liquids has been studied in other studies as well. The aim of this study was to present a unique new type of deep eutectic solvent of the fourth type, which is rarely used in the electroplating process. It was produced from mixing lithium chloride (LiCl) with 1,3 propanediol with a molar ratio of 1:4. In addition, the use of lithium chloride instead of choline chloride, in spite of their having the same anion, is due to the small size of Li^+ compared with the choline ion, which has a strong influence on physical properties and will show its effect on the density and conductivity of DEs. Also investigated the effects of various additives on electrolytic deposition of copper at high temperatures.

2. Materials and methods

2.1. Chemicals and materials

The following chemicals were purchased from Sigma Aldrich and used without further purification: 1,3-propanediol ($C_3H_8O_2$, > 99.8%) and lithium chloride (LiCl, ≥ 98%). using hydrated copper chloride (Aldrich, $CuCl_2.2H_2O$, ≥ 98%). Triton x100 (Tx100, 0.02 M) (Aldrich - Sigma, 99%) and Tween 20 (Tw20, 0.01 M) (Aldrich – Sigma ≥ 99.5%) were the additives used.

2.2. Preparation of new deep eutectic solvent DES

Lithium chloride LiCl and 1,3- propanediol are mixed in a molar ratio of 1:4 to create novel DESs. This is done by dissolving 10 g of lithium chloride with 144.76 g of 1,3 propanediol on a hot plate at 50 °C for two hours while stirring constantly until a homogenous, colorless liquid solution is produced (**Figure 1**).

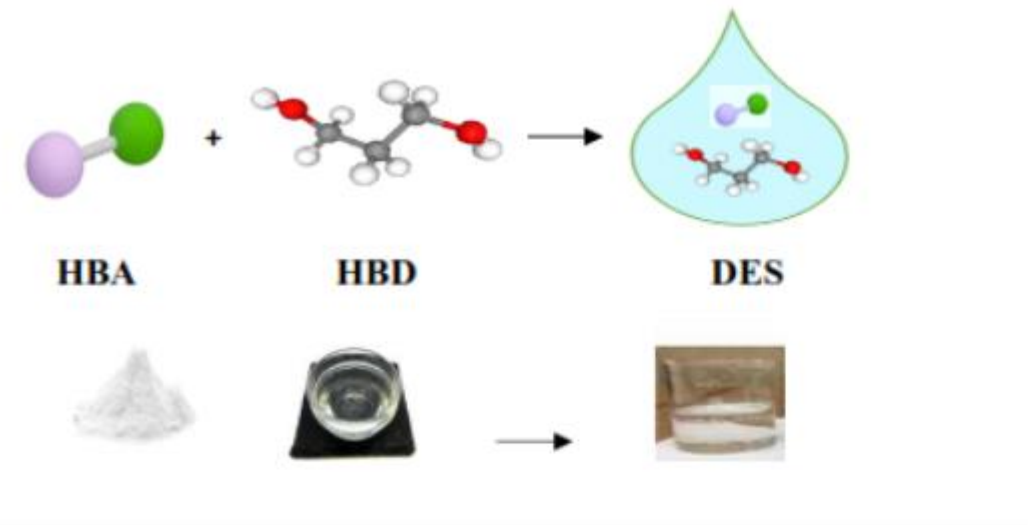


Figure 1. Shows the new DES preparation.

2.3. Copper electrochemical analysis

The GPES2 software controlled an IVIUM potentiostat / galvanostat used for the cyclic voltammetry (CV) (**Figure 2**). The cell used in these tests consisted of three electrodes: an Ag wire for the reference electrode, a Pt (1.3 mm) working electrode, and a Pt flag counter electrode. Every experiment's working electrode was cleaned with H_2O distilled water after being polished with 0.05 µm γ-alumina paste. Every CV to $CuCl_2.2H_2O$ cyclic voltammograms, with or without addition, was carried out in DESs with a potential window of 1.0V to −1.0V and at the same temperature (80 °C). Changes were done to the scan rates (10–60 mVs^{-1}), concentrations (0.1-0.9M), temperatures (30–80 ºC),and use different concentrations of additives (0.02M Tw20, 0.01M TX100, %10H_2O). Increasing the concentration of $CuCl_2{\cdot}2H_2O$ in the solution not only affects the increase in reduced peak currents but also influences the oxidation (stripping) process during the cyclic voltammetry measurement. This happens because higher concentrations of $CuCl_2{\cdot}2H_2O$ lead to a greater presence of $[CuCl_4]^{2-}$ complexes in the solution, which aid in copper deposition. During the reverse scan in cyclic voltammetry, these complexes or the deposited copper can be oxidized back into ions, resulting in additional or more prominent stripping peaks in the voltammogram.

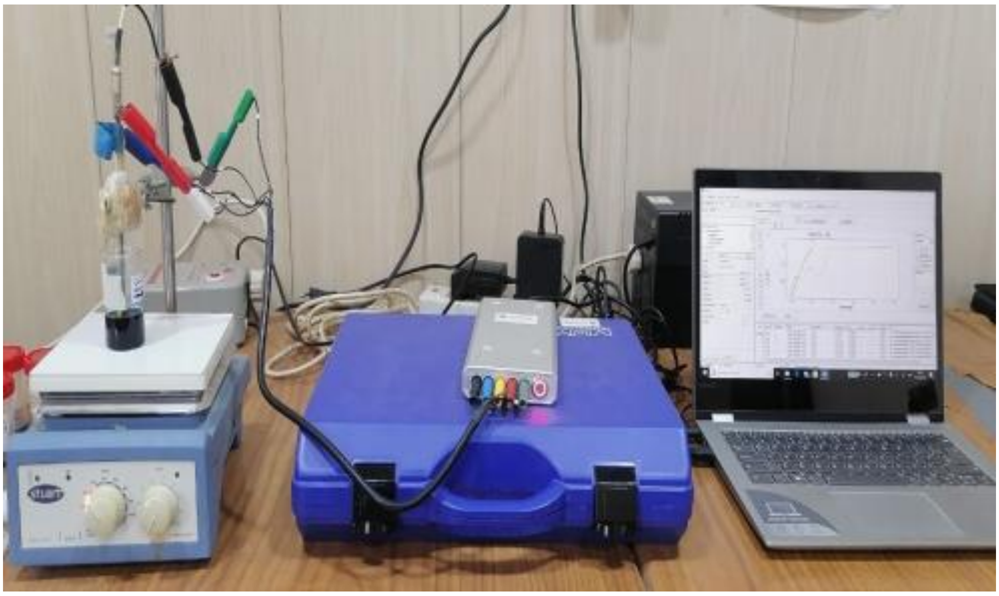


Figure 2. Shows Electrochemical of $CuCl_2.2H_2O$ in DES.

2.4. Electroplating of copper chloride $CuCl_2.2H_2O$

Using 0.3 M $CuCl_2.2H_2O$ without or with additives, copper electrochemical coatings were created in DES at 80 °C for two hours at a current of 0.012 mA cm^{-2} on a nickel substrate (which acts as the cathode electrode in the copper deposition process). A 0.87 M aqueous solution of ammonium persulfate $(NH_4)_2S_2O_8$ and a 0.2 M acid solution of sulfuric H_2SO_4 were used to etch the nickel Ni substrate before the experiment. It was then thoroughly washed with H_2O distilled water and allowed to dry. In contrast, the anode utilized in this work was a 50 x 60 mm Ti mesh covered with iridium oxide (**Figure 3**).

Figure 3. Shows electroplating of $CuCl_2.2H_2O$ in DES.

2.5. Surface analysis technique

Scanning electron microscopy (FESEM) was used to describe the surface. Additionally, atomic force microscopy (AFM) is used to determine the shape and nucleation of metal deposits.

3. Results and discussion

3.1. Cyclic voltammetric (CV) studies

As shown in **Figure 4**, the cyclic voltammograms (CVs) of pure deep eutectic solvent (DES) and DES with 0.3 M $CuCl_2.2H_2O$ are compared. We notice specifically in this figure that the oxidation-reduction behavior of these two systems is quite different. The CVs of DES with an infusion of $CuCl_2.2H_2O$ at 0.3 M exhibit two peaks. These peaks give an idea about the redox process of copper ions. The initial peak is the change of Cu^{2+} to Cu^+, whereas the second peak denotes the conversion of Cu^+ to metal Cu (Cu^0). Such behavior is seen for copper redox transitions, which indicates the electrochemical activity of copper in the DES medium. In contrast, the CVs of pure DES have no peaks that can be detected. This shows that the pure DES did not exhibit any meaningful redox activity under these experimental conditions. The flat profile of pure DES CVs indicates that there are no electroactive species oxidizing or reducing in the solvent only. A three-electrode system was used for the experiments. For all measurements, the scan rate was 30 mV/s, and the temperature was kept at 80 °C. To avoid misinterpretation of the CVs of both systems, these experimental parameters were kept constant. The difference between the electrochemical behaviors of pure DES and DES with 0.3 M of $CuCl_2.2H_2O$ shows the role of the copper ions in redox. The importance of the metal salt additives for the turning of electrochemical properties of DES was paid attention to in the analysis showing how it would affect the systems.

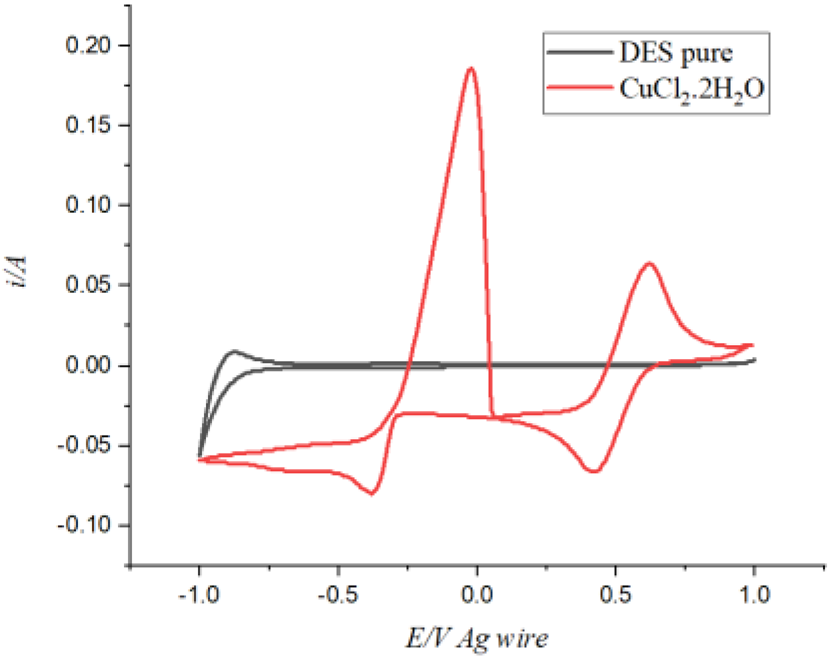


Figure 4. Cyclic Voltammograms of novel DES (Black Line) and 0.3 M $CuCl_2.2H_2O$ in DES (Red Line) both at 80°C, recorded at a scan rate of 30 mV/s, Using a 1.3 mm Pt disc working electrode, Pt flag counter electrode, and referenced against Ag wire.

3.2. Effect of concentrations on the C.Vs of copper Cu^{+2} with deep eutectic solvent

In this work, as seen in **Figure 5**, the usage of novel DES (LiCl + 1,3- propanediol) to electrodeposit $CuCl_2.2H_2O$ at various concentrations (0.1, 0.3, 0.5, 0.7 to 0.9 mole/L) was conducted. The cyclic voltammetry of various concentrations of $CuCl_2.2H_2O$ in this DES at elevated temperatures (80 ºC) is an interesting research area. The voltammetry was completed at 30 mV/s and in the potential range of 1.0 V to -1.0 V, using a platinum flag as a counter electrode; working electrode (platinum Pt) (1.3 mm) and the reference electrode (Ag wire) The increase in $CuCl_2{\cdot}2H_2O$ concentration in the electrolyte led to higher reduction peak currents, as illustrated in **Figure 2**. This increase is attributed to the elevated levels of $[CuCl_4]^{2-}$ in the solution, which facilitated the deposition of a significant amount of copper (Cu). Conversely, lower concentrations of $[CuCl_4]^{2-}$ had the opposite effect, resulting in reduced peak currents. The abundance of $[CuCl_4]^{2-}$ and the limited availability of chloride anions (Cl^-) at the surface reduced the energy required to reduce Cu^{2+} ions. Moreover, the high concentration of $CuCl_2{\cdot}2H_2O$ promoted the formation of additional stripping peaks, as shown in **Figure 2**, thereby enhancing the Cu deposition potential[20,25].

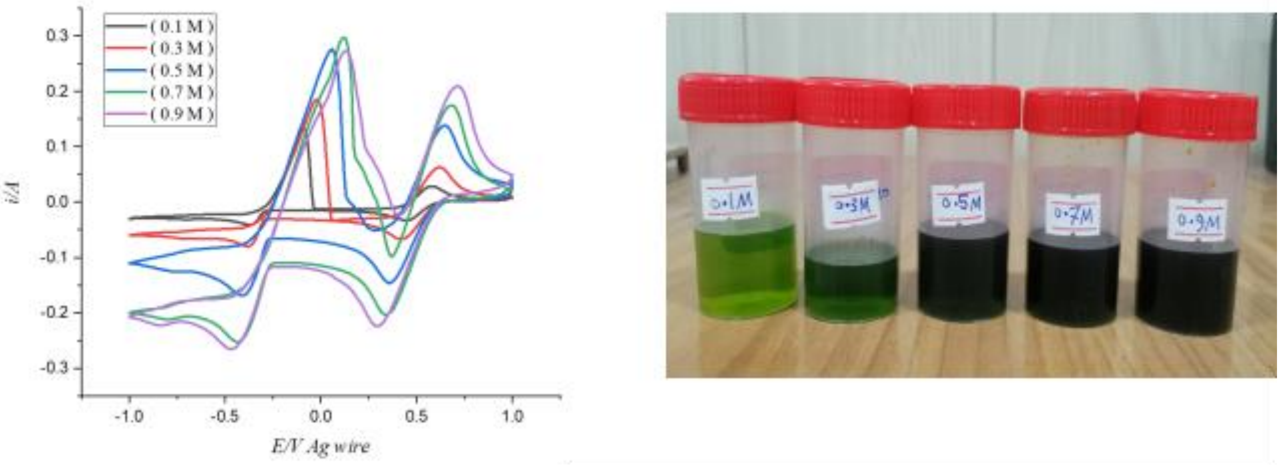


Figure 5. Effect of Concentration on $CuCl_2.2H_2O$ in 25 ml of $CuCl_2.2H_2O$ in novel DES (1LiCl: 4 (1,3-propanediol).

3.3. Effect of scan rate on the CVs of copper Cu^{+2} with Deep Eutectic Solvent

Cyclic voltammograms taken at 80 °C (Ag wire as reference electrode, Pt flag as counter-electrode, 1.3 mm Pt disc as working electrode) were performed with a range of scan rates of 10 to 60 mV/s in periodic square wave for a 0.3 M solution of $CuCl_2.2H_2O$ in deep eutectic solvent (DES). A voltage range of -1 to 1 was applied with a direction inversion to -1, after which it was powered back to the start. It can be noted in **Figure 6** that the greater the scan rate, the greater the copper deposition and deeper the peak of Cu became with time. This connotes the limiting factor of the kinetics of the electrode reactions due to the scan rate. During rapid scan rates, there was a noticeable enhancement in oxidative but little in cathodic currents; even oxidation was dominating. But because there was a greater amount of Cu deposition, copper was able to develop in greater efficiency. This suggests that, at higher frequencies, the migration and accumulation of DES copper at the electrode occur more readily compared to lower frequencies[26]. It is believed that the stronger deposition efficiency at faster scan rates is due to the diminished demand of diffusion-controlled processes, resulting in more favorable kinetics for electrodeposition. These effects indicate how important scan rate has in controlling the efficiency and rate of Cu deposition during the cyclic voltammetry of $CuCl_2.2H_2O$ in DES[27].

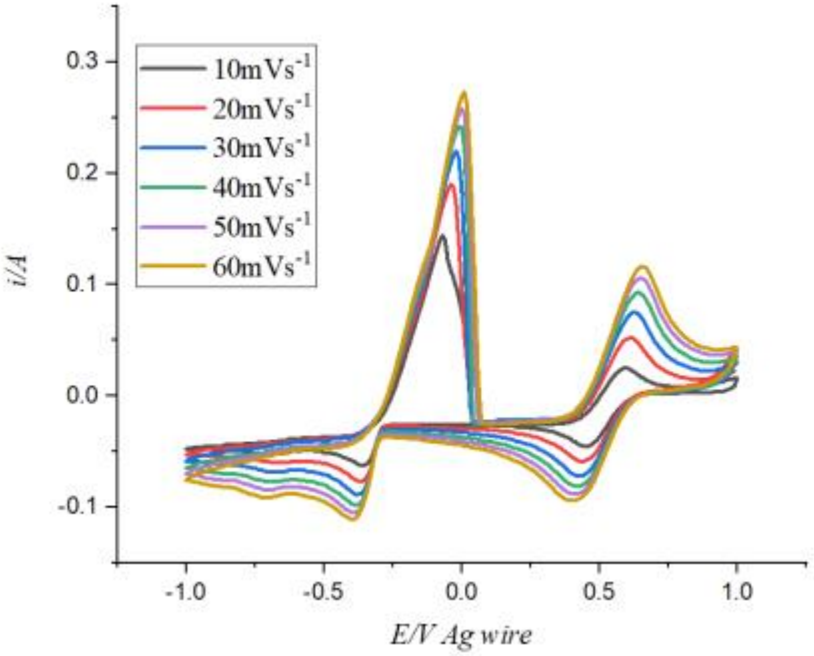


Figure 6. Effect of scan rate on 0.3 M $CuCl_2.2H_2O$ in 25ml of novel DES (1LiCl: 4 (1,3- propanediol).

3.4. Effect of temperature on the C.Vs of Cu^{+2} with deep eutectic solvent

Using a new deep eutectic solvent (DES) comprising LiCl and 1,3-propanediol, the CVs of Cu electrodeposition from 0.3 M $CuCl_2.2H_2O$ diluted in a potential solvent were shown in **Figure 7**. CVs were acquired across the temperature range of 30 to 80 °C and at a scan rate of 30 mV/s. The graphic illustrates how the decrease peak shifts anodically with rising temperatures. This is most likely caused by the DES's inherent viscosity at higher temperatures, which permits Cu to deposit at a lower potential[28]. By decreasing the specific adsorption of free chloride ions (Cl^-) on the electrode surface, high temperatures can encourage the reduction of copper (Cu). This lowers the activity and mobility of chloride ions while raising the concentration of $[CuCl_4]^{2-}$ near the electrode[29]. This speeds up the rate of Cu deposition by preventing deep eutectic solvent (DES) cations from sticking to the electrode surface.

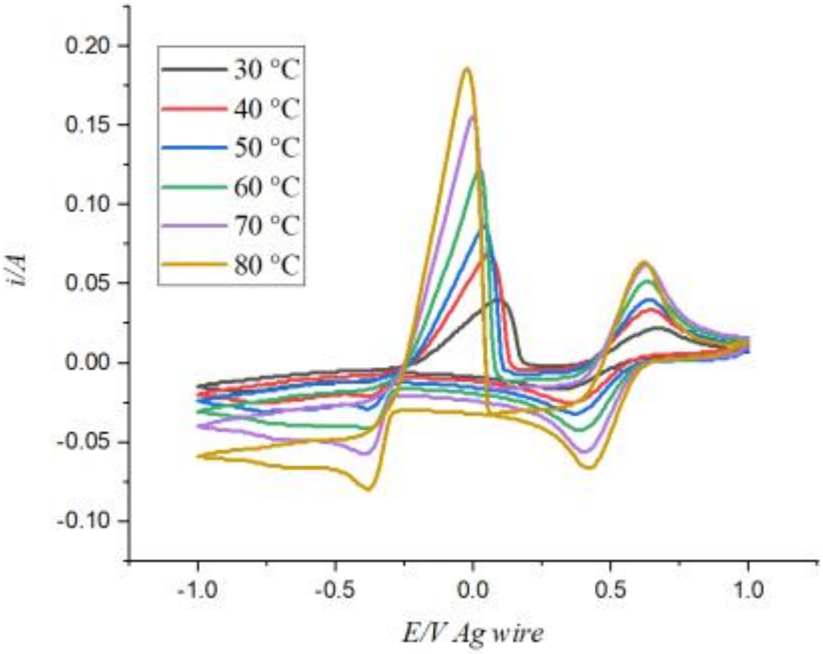


Figure 7. Effect of temperature on 0.3 M $CuCl_2.2H_2O$ in 25 ml of novel DES (1LiCl: 4 (1,3 -propanediol).

3.5. Effect of additions on the voltammetry behavior of Cu^{+2}

The results of cyclic voltammetry (CV) on 0.3 M $CuCl_2.2H_2O$ in DES at 80 °C with varying additive concentrations, such as (a) Triton X100, (b) Twien20, and (c) 10% H_2O, are displayed in Figure 8. Cyclic voltammograms (CVs) were performed at a rate of 30 mV/s using a platinum flag counter electrode, an Ag wire reference electrode, and a working electrode (Pt 1.3 mm). The impacts of various additives on the new DES's electrochemical performance were only investigated at 80 °C. **Figure 5** illustrates this point. The range of the drop peak is -1.0V to 1.0V. Therefore, the voltammetry test demonstrates that the transport of ions to the expanding three-dimensional mass of a new phase controls the process. When Triton X100 was added, the reduction of unique DES peaks also increased dramatically; however, when Tween 20 was used, the peak decline dropped. This is an indication of copper Cu deposition. Additions to the electrolyte slow down copper plating at relatively negative potentials and enhance the likelihood of the transition from abrupt to gradual nucleation. This explains the discernible smoothing effect on the shape of the plating copper and validates their catalytic activity. This validates their catalytic activity and explains the discernible smoothing effect on the morphology of the plating copper.

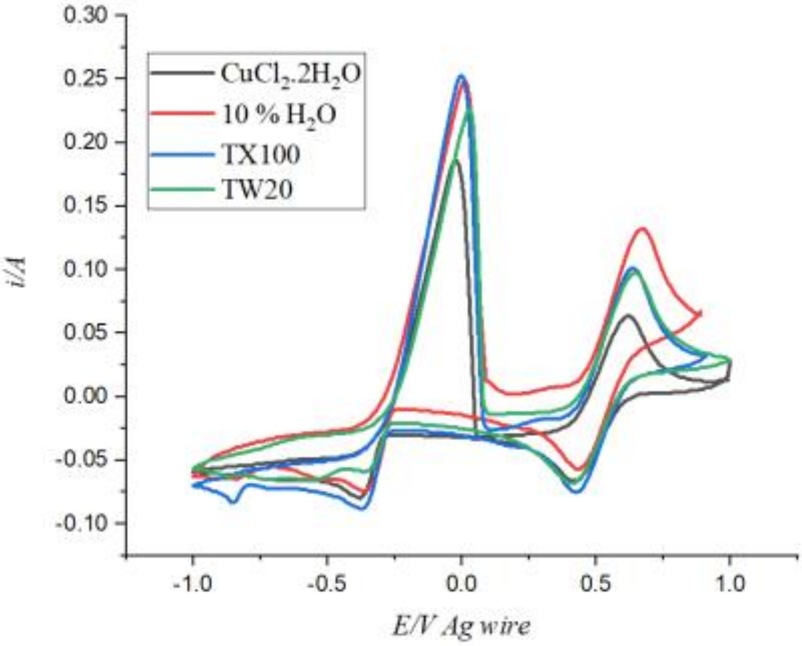
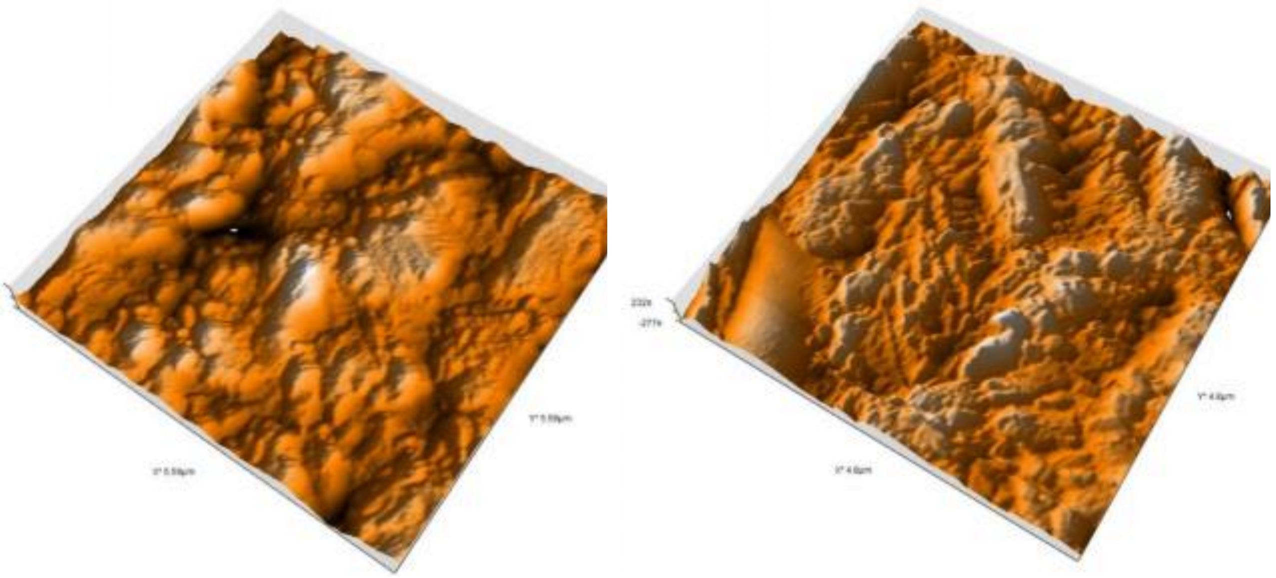


Figure 8. Cyclic Voltammogram C.V at 80°C of 0.3 M $CuCl_2.2H_2O$ in a 1:4 Molar Mixture of Lithium Chloride LiCl + 1,3-Propandiol using different amounts of additives 0.02 M TritonX100, 0.01 M Tween20 and 10% H_2O.

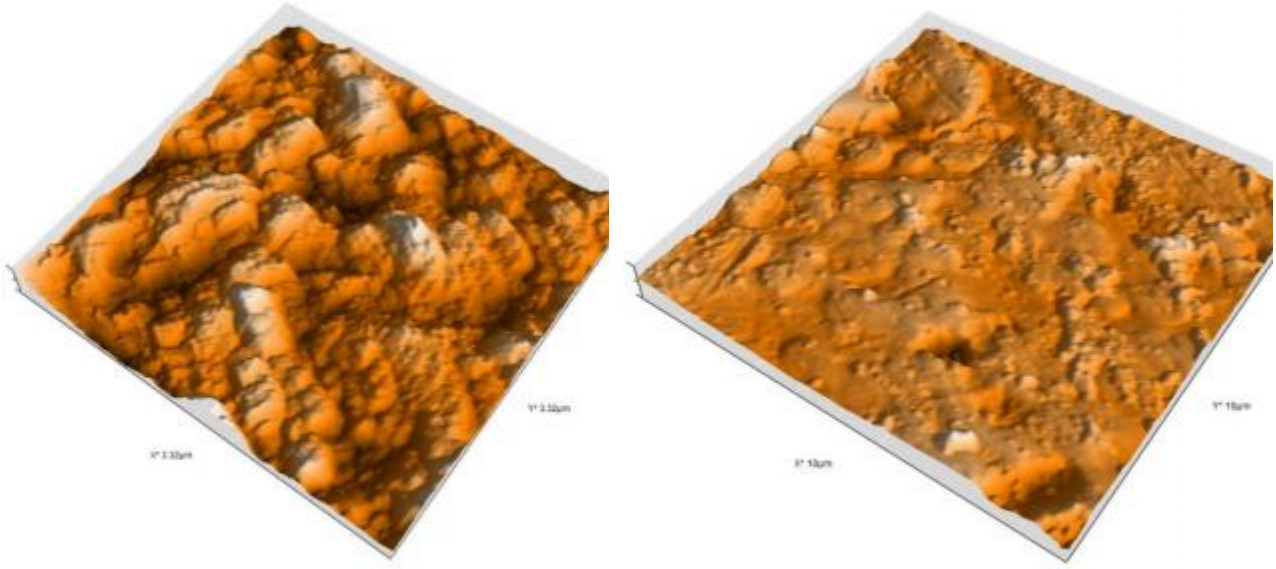
3.6. Surface characterization

3.6.1. Atomic force microscopy (AFM) study

AFM was used to analyze the roughness and terrain of Cu growth (0.3 M $CuCl_2.2H_2O$) from new DES at 80 °C for two hours at 0.012 mA cm^{-2} on a nickel substrate, both with and without additions. according to **Figure 9**. The addition of additives like 10% H_2O, 0.01 M Tween20, and 0.02 M Triton X 100 causes a dramatic alteration in the morphology of copper coatings. Without the additives, the deposited Cu displayed an extremely high degree of roughness. However, the presence of additives results in a much smoother and more uniform surface morphology. where the addition of chemicals resulted in a decrease in roughness. The surface of the electrodeposited Cu layer of the additions-free electrolyte was clearly rougher and had bigger grain sizes.



(a) **(b)**



(c) (d)

Figure 9. AFM Images for the plating from 0.3M $CuCl_2.2H_2O$ in novel DES with and without additions. The deposition was (a) DES + $CuCl_2.2H_2O$ (b) 10% H_2O (c) 0.02 M TritonX100, (d) 0.01 M Tween20.

3.6.2. Field emission-scanning electron microscopes (FE-SEM) study

Field emission-scanning electron microscopy (FESEM) was used in this investigation to examine the impact of additives on the surface morphology and deposition quality for 0.3 M $CuCl_2.2H_2O$ in the innovative DES electrolyte at 80 °C. The FESEM images, presented in Figure 10, illustrate the differences between the copper deposits obtained with and without additives. In all experiments, a mild nickel substrate was used as the cathode, while 10% H_2O, 0.01 M Tween20, and 0.02 M TritonX100 were the three additives examined. **Figure 10-a** depicts the morphology of the Cu deposit obtained from an additive-free electrolyte, where the surface exhibits a high degree of roughness and irregularly sized Cu grains, indicating uncontrolled nucleation and non-uniform deposition. In contrast, the addition of 10% H_2O (**Figure 10-b**) results in a smoother, more uniform deposition, significantly reducing surface roughness and promoting improved nucleation. Triton X100 (**Figure 10-c**) enhances the compactness and uniformity of the deposited layer, producing a well-leveled Cu film with improved brightness, suggesting its role as a leveling agent. Similarly, Tween20 (**Figure 10-d**) facilitates the formation of a well-structured, homogeneous copper layer with reduced surface defects and more refined grain size. These findings confirm that the choice of additives greatly influences copper deposition, affecting grain refinement, surface smoothness, and coating brightness. The observed improvements in coating morphology with Triton X100 and H_2O validate their effectiveness as brighteners and leveling agents in Cu electrodeposition. This aligns with the voltammetric behavior observed in DES electrolytes containing these additives, further supporting the hypothesis that controlling additive composition enhances the overall quality of Cu films[1-5,30].

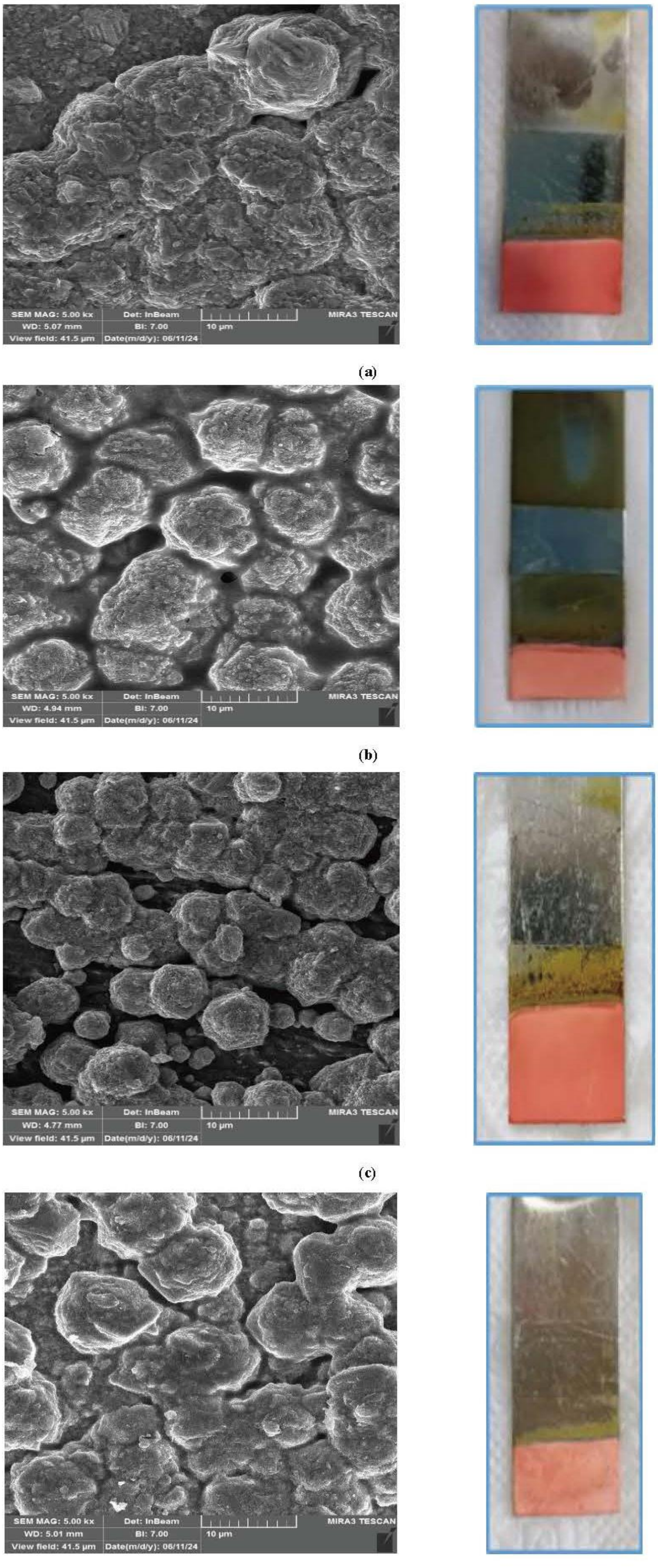


Figure 10. Optical Photographs, SEM Morphologies, of 0.3M$CuCl_2.2H_2O$ plating from novel with and without additions. Images (a) without additives, (b) 10% H_2O, (c) 0.02 M TritonX100, (d) 0.01 M Tween20.

3.7. Physical Properties

3.7.1. Density

Figure 11 shows that the density of the copper chloride solution ($CuCl_2.2H_2O$ M + DES) declines with increasing temperature. This is because when temperature rises, free volume rises and viscosity decreases. The addition of Tx100, Tw20, and 10%H_2O will increase the density of the copper chloride solution. The density of the additives in the copper chloride solution ($CuCl_2.2H_2O$ + DES + TW20, TX100, 10% H_2O) decreases as the temperature rises, as seen in **Figure 9**.

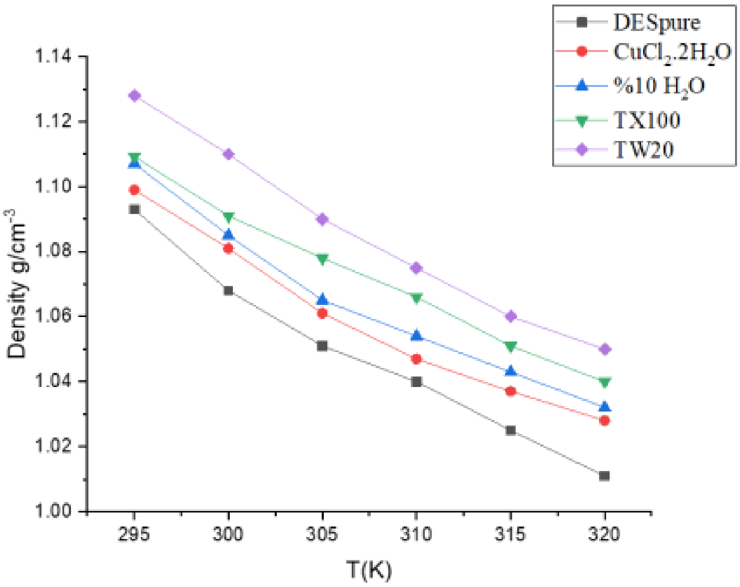


Figure 11. The density of 0.3 M $CuCl_2.2H_2O$ in 25 ml of DES as a function of temperatures with various additive.

3.7.2. Conductivity

The conductivity of the copper solution and its additions was also evaluated. $CuCl_2.2H_2O$ conductivity in DES is plotted against temperature in **Figure 12**, both with and without additions. Because of a reduction in the solutions' viscosity, we observed a rise in conductivity with the temperature rise. Conductivity is decreased when additives such as Tween20 and TritonX100 increase the viscosity of the Cu solution. By decreasing the viscosity of the copper Cu solution with additional additions of 10% H_2O, conductivity is increased. Is represented by **Figure 12**.

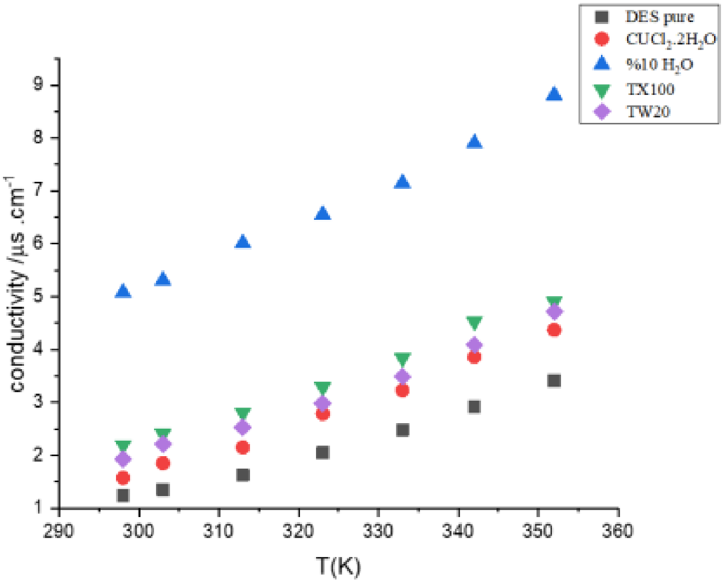


Figure 12. The conductivity of 0.3 M $CuCl_2.2H_2O$ in 25 ml of novel DES as a function of temperatures with various additives.

4. Conclusions

The aim of this work was to develop a new deep eutectic solvent (DES) and apply it to copper electrodeposition on nickel (Ni) substrate. The study looks into how additives like H_2O, TritonX100, and Tween 20 affect Cu electrodeposition. Numerous additives have had an impact on copper's Cu electrochemical coating. This experiment demonstrated that the additions greatly increased the Cu deposition in this system when compared to the identical solution without the addition. The addition to the Cu-containing solution caused significant changes in the cyclic voltammetry for Cu. In the copper cyclic voltammetry studies, we found that TritonX100 and H_2O both cause the reduction peak to increase. According to AFM studies, when electrodeposition is performed with the additive present in the electrolyte, certain additives, such as TritonX100 and H_2O, have outstanding effects on enhancing the coating for Cu. The ensuing deposition has demonstrated a significant decrease in the crystallites' surface roughness and grain sizes. According to the FE-SEM results, TritonX100 and H_2O gave Cu film on nickel (Ni) substrate strong deposition and adherence. Hard adherent Cu coating was generated by the deposition occurring in novel DES at 80 °C, and the use of additives is beneficial for the final coating.

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Author contributions

Authors contributed equally to the manuscript

Conflict of interest

The authors declare no conflict of interest

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