



## Silver Recovery from E-Waste Printed Circuit Board Using Binary and Ternary Deep Eutectic Solvents

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### Abstract

Printed Circuit Boards (PCBs) are essential components of electronic devices containing valuable silver metal. Using sustainable methods, silver recovery from electronic trash, like PCBs, demonstrates excellent promise. This research's objective is to determine the optimum leaching time and solid-to-liquid (S/L) ratio for extracting silver from PCB using deep eutectic solvent (DES) composed of choline chloride and glycerol (glyceline DES). The binary DES's leaching performance was then compared to choline chloride, glycerol, and citric acid ternary systems. Fourier Transform Infrared Spectroscopy (FTIR) was carried out to analyze the bond interactions. X-ray fluorescence spectrometry (XRF) was employed to determine the PCB's metal concentration prior to and after the leaching process. Ternary DES was viscous, colorless, stable for 60 days, and less acidic than binary DES, with a 1.21 g/mL density. FTIR peak broadening and shifting indicated the formation of a new hydrogen bond and proved a successful synthesis of ternary DES. XRF result showed that PCB's initial silver metal content was 2.32%. The optimal silver leaching from PCB using glyceline DES was achieved after 16 hours, with a 1/20 solid-to-liquid ratio. Ternary DES demonstrated a silver leaching efficiency of 93.65%, surpassing 86.77% of glyceline. Ternary DES synthesized in this study has the potential to serve as an efficient and environmentally friendly solvent for extracting silver from PCB, providing a sustainable approach to managing electronic waste.

### 1. Introduction

The amount of electronic waste is projected to increase from around 53.6 million tons in 2019 to 74.7 million tons by 2030; however, only 17.4% of the entire amount is recycled [1]. The issue of electronic waste is also addressed in the United Nations Sustainable Development Goals (SDGs), specifically in Goal 11, "Make cities and human settlements inclusive, safe, resilient, and sustainable," and Goal 12, "Ensure sustainable consumption and production patterns," which aims to reduce waste accumulation and ensure proper management of chemicals through prevention, reduction, improvement, and reuse [2]. According to the United Nations Institute, approximately 51% of the generated electronic waste is attributed to Printed Circuit Boards (PCBs) [1]. PCBs are essential components in electronic devices, making up around 20–30% of mobile

phones, 20% of computers, and approximately 10% of televisions [3]. One method to recycle PCB involves extracting and recovering precious metals, such as silver [4]. Silver metal holds a high economic value, costing \$528,558 per ton [5]. The substantial value of this element lies in its essential function as a conductor and electrode, thanks to its exceptional electrical conductivity [6]. Hence, the recovery of silver metal from electronic waste such as PCBs shows excellent potential.

The conventional methods utilized for silver metal recovery from PCB waste generally involve pyrometallurgy and hydrometallurgy processes. Pyrometallurgy is the method of extracting metals using smelting, combustion, pyrolysis, and molten salts [7]. However, it should be noted that pyrometallurgical processes are costly and environmentally unfriendly [8].

Hydrometallurgy is the metal recovery method by using acid or alkaline hydrous-based solutions, for instance, cyanide, halide, thiourea, and thiosulfate, to dissolve solid compounds hydrometallurgy. The procedure necessitates a complex pretreatment with multiple chemicals, making it a time-consuming and expensive method [9]. An alternative approach called solvometallurgy has been proposed as a potential choice. Solvometallurgy involves metal extraction from various sources, including ores, industrial process residues, production residues, and municipal waste, using non-aqueous or low-water-content solvents. These non-aqueous solvents can include molecular organic solvents, ionic liquids, and deep eutectic solvents (DES), as well as inorganic solvents such as liquid ammonia, concentrated sulfuric acid, or supercritical carbon dioxide [10].

Several solvents have been employed to extract silver from PCB, including inorganic acid solvents using caustic reagents, but these can be hazardous to both workers and the environment [11]. Furthermore, organic acid solvents like malic acid or oxalic acid have been found to exhibit lower efficiency and require additional additive compounds to facilitate metal leaching [12]. The use of ionic liquids (ILs) as alternative options to traditional organic solvents was initially proposed in 1914 to tackle environmental issues [13]. However, it is worth noting that ILs exhibit poor biodegradability, biocompatibility, and shelf life. ILs are also fancy, toxic, and complicated to be synthesized. Consequently, deep eutectic solvents (DES), invented in 2003, share numerous advantages of ILs but are considerably safer, more economical, and easy to manufacture [14, 15]. DES has several applications, including metal electrodeposition, leaching, biocatalytic activities, and organic synthesis [16]. DES is anticipated to function as an eco-friendly solvent that can reliably and efficiently leach silver from PCB waste.

Aldhafi [17] reported research on silver metal leaching from PCB employing DES glyceline consisting of choline chloride as the hydrogen-bond acceptor (HBA) and glycerol as the hydrogen-bond donor (HBD). This study investigated the effect of temperature on the silver leaching, resulting in an 84.42% yield at a maximum temperature of 120°C. However, the study has not yet examined the influence of time and solid-to-liquid (S/L) ratio on the silver extraction capability. Therefore, this study continues the previous work to determine the optimum time and S/L ratio for leaching silver from PCB waste using the DES glyceline.

Recently, significant progress has been made in developing ternary deep eutectic solvents (DES) composed of three combinations of HBA and HBD. In the recent work, Kadhom *et al.* [18] synthesized two types of ternary DES: choline chloride-urea-glycerol and choline chloride-malic acid-glycerol. The research findings show a significant correlation between variations in density, thermal stability, and other characteristics with the molar ratio of components in the ternary combination. Ternary DES has been applied in various domains, including lignin dissolution [19], wheat straw decomposition [20], and CO absorption [21]. Additionally,

ternary DES possesses a distinct advantage in metal leaching, as demonstrated in research by Huang *et al.* [22]. It demonstrates ternary DES's better efficiency than binary DES in extracting cobalt metal from discarded lithium-ion batteries.

To the best of the author's knowledge, a study regarding the leaching efficiency of silver from PCB using a deep eutectic solvent is comparatively scarce. Hence, the objective of this study is to continue the previous research of Aldhafi [17] by investigating the optimal time and S/L ratio for silver leaching using glyceline binary DES. Subsequently, an attempt is made to utilize ternary DES by adding citric acid to glyceline binary DES to recover silver from PCB samples. Citric acid as an HBD was chosen based on studies conducted by Peeters *et al.* [23], which report that citric acid as an HBD has a higher efficiency in leaching cobalt metal than other HBDs. This is also a preliminary study to investigate the effectiveness of ternary DES compared to widely used binary DES glycelin for leaching silver leaching from PCB.

## 2. Experimental

### 2.1. Equipment and Materials

The equipment used in this research included glassware, an analytical balance, a hotplate magnetic stirrer, a desiccator, and a thermometer. Characterization was conducted using a Fourier Transformation Infrared (FTIR) spectrometer (Prestige 21 Shimadzu) and an X-ray fluorescence (XRF) spectrometer (EDAX Orbis Micro-XRF). The materials and chemicals used in this study included Printed Circuit Boards (PCBs) from discarded computers, choline chloride (LOBA Chemie, solid, MW: 139.63 g/mol), glycerol (LOBA Chemie, Purity 99.5%, liquid, MW: 92.09 g/mol), and monohydrate citric acid (Merck, solid, MW: 210.14 g/mol).

### 2.2. PCB Sample Preparation

The sample preparation of PCB was performed by separating components such as resistors, capacitors, diodes, and other components attached to computer PCB waste. Subsequently, the PCB boards were crushed and heated in a furnace at 600°C. After heating, the samples were pulverized and sieved to transform them into a powdered form. The PCB sample powder was analyzed using an XRF spectrometer (ORBIS EDAX AMETEK) with the energy element range measured at 40 kV, 355 µA under vacuum for 90 seconds to calculate the initial amount of silver metal in the PCB before the leaching process. Figure 1 presents a schematic representation of the printed circuit board (PCB) preparation process.

### 2.3. Synthesis of Glyceline DES and Ternary DES

The glyceline DES was synthesized by mixing choline chloride and glycerol in a 1:2 molar ratio [17]. The glyceline DES was characterized for its physical properties to confirm its conformity with the DES prepared by Aldhafi [17]. The ternary DES synthesis was performed by mixing choline chloride, glycerol, and citric acid in a 7:10:3 molar ratio [24]. The ternary DES was characterized by analyzing the chemical properties using an FTIR spectrometer (Prestige-21 Shimadzu) using the

wavenumber range from 400–4000 cm<sup>-1</sup> with a resolution of 1 cm<sup>-1</sup>).

#### 2.4. Optimization of Leaching Parameters

The optimization of silver leaching parameters from PCB using glyceline DES was performed by varying time and solid-to-liquid (S/L) ratio variables. Leaching time variations were carried out at five different time intervals: 4, 8, 16, 24, and 32 hours with a fixed leaching temperature, solid/liquid ratio, and stirring rate of 120°C, 3/20, and 500 rpm respectively.

Solid-to-liquid (S/L) ratio variations (g/mL) were tested with five ratios: 0.5/20, 1/20, 2/20, 3/20, and 4/20 with a constant leaching temperature of 120°C, stirring rate of 500 rpm, and the optimum leaching time determined from leaching time optimization. The results of each leaching optimization were analyzed using an XRF spectrometer.

#### 2.5. Leaching with Ternary DES

The leaching process of PCB waste utilizing ternary DES was carried out under optimal conditions (time, temperature, and S/L ratio) using glyceline DES, followed by after-leaching-residue analysis using an XRF spectrometer. This phase aimed to analyze the effect of adding citric acid to glyceline DES, resulting in the formation of ternary DES (Figure 1), and to evaluate the efficiency of glyceline DES with ternary DES in extracting silver from PCB waste. The leaching percentage was determined using Equation (1).

**Table 1.** Percentage of metals in the research PCB sample

Element	Weight percentage
Copper (Cu)	33.27%
Aluminum (Al)	8.18%
Tin (Sn)	5.62%
Silver (Ag)	2.32%
Lead (Pb)	1.32%

$$\% \text{ Extraction} = \frac{m_f}{m_i} \times 100\% \quad (1)$$

where,  $m_f$  is the final mass of silver in PCB residue after leaching (g), and  $m_i$  is the initial mass of silver in PCB (g).

### 3. Results and Discussion

#### 3.1. PCB Waste Sample

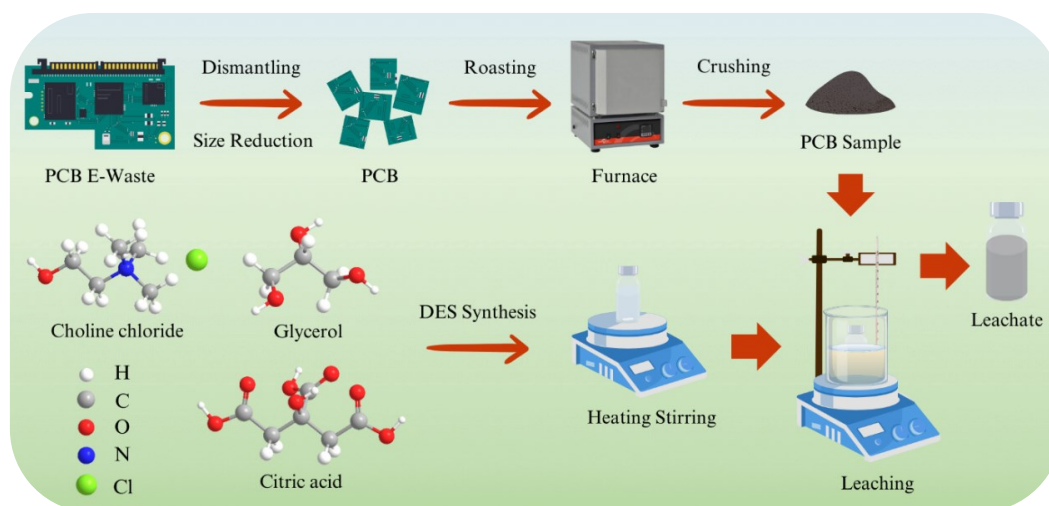
XRF spectrometer was used to measure the initial metal's concentration before the leaching procedure. From the analysis, the most dominant metal content in the sample was copper at 33.27%, followed by aluminum at 8.18%. In particular, the concentration of silver in PCB was 2.32%. These results align with the metals composition commonly found in PCBs, as previously studied by Vats and Singh [25]. The total metal content analysis in the PCB sample is presented in Table 1.

#### 3.2. Glyceline DES Synthesis

The DES glyceline synthesis involved mixing choline chloride and glycerol in a 1:2 molar ratio. No solid formation or color change was observed after 60 days of storage in a desiccator, indicating the DES stability and the successful completion of synthesis [26]. This DES appeared as a viscous and colorless liquid with a 1.1407 g/mL density. This physical property was in agreement with Aldhafi [17]'s findings, showing a value around 1.1378 g/mL and within the range of values recorded in other literature (1.1495 g/mL) [27]. Additionally, glyceline DES was subjected to pH testing and showed an acidity level of 5.

#### 3.3. Optimization of Silver Leaching Using Glyceline DES

The study focused on optimizing the silver leaching from PCB waste by varying time and solid-to-liquid (S/L) ratio parameters. This leaching optimization aims to determine optimal conditions for silver leaching by glyceline DES to maximize metal extraction efficiency [22]. Additionally, understanding the best leaching conditions improves the efficiency, cost-effectiveness, and environmental sustainability of metal extraction [28].



**Figure 1.** Schematic illustration of the PCB preparation and the synthesis of glyceline DES and the ternary DES

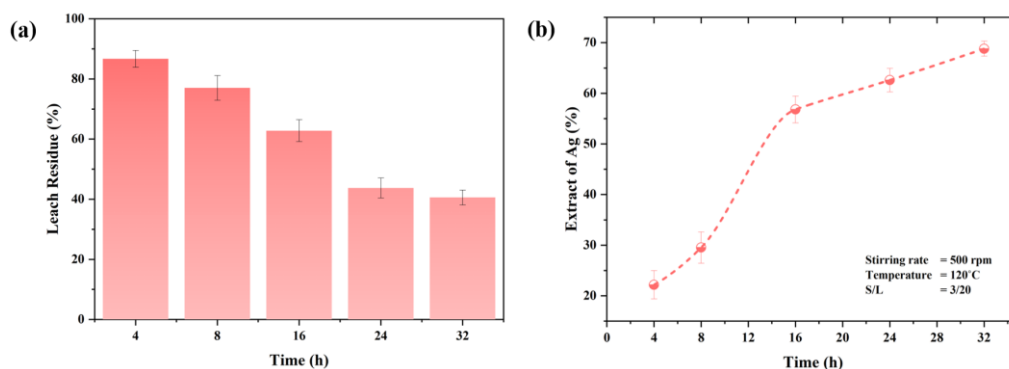


Figure 2. Leaching time optimization results in (a) the percentage of residue produced after leaching and (b) the percentage of extracted silver

### 3.3.1. Influence of Leaching Time Parameter

Leaching time optimization was carried out to enhance metal extraction efficiency by minimizing prolonged durations that result in increased energy consumption. Generally, metal leaching efficiency increases with extended leaching duration until it peaks, signifying solvent saturation [29, 30]. The XRF spectrometer results, depicted in Figure 2a, indicate that increasing the leaching period leads to decreased residue generated and more dissolved metals in the DES.

Figure 2(b) shows that the proportion of silver metal extracted increases significantly until the leaching time is 16 hours, then levels off from 24 to 32 hours. This phenomenon might be attributed to the decreased or near-saturated capacity of the glyceline DES to dissolve metals from the PCB [29]. As a result, the metal dissolution process occurs slowly and leads to incomplete metal extraction. This current study selected 16 hours as the maximum leaching time for the following investigation. The selection of a 16-hour period is supported by a graph demonstrating a significant rise in silver metal extraction, up to 56.80%. Moreover, it is important to consider that extending the leaching time may increase energy consumption and diminish the effectiveness of DES as a green solvent.

### 3.3.2. Influence Solid-to-Liquid (S/L) Ratio Parameter

The solid-to-liquid (S/L) ratio was optimized to achieve standard conditions that yield the highest leaching efficiency [31]. A decreased S/L ratio typically enhances metal leaching due to the increased contact area between the solid and liquid phases [32]. This finding demonstrates that the residue amount decreases as the

S/L ratio drops, as depicted in Figure 3(a). In addition, Figure 3(b) displays a consistent upward trend in the percentage of silver metal recovered until the S/L ratio reached 1/20, which can be attributed to improved mass transfer between solid and liquid interphase [29].

This choice was based on a substantial increase in silver metal extraction, reaching 86.77% at the 1/20 ratio. Selecting a 0.5/20 S/L ratio resulted in a slight increase (from 86.77% to 90.56%) but required a larger quantity of glyceline DES (40 mL of DES (0.5/20) compared to 20 mL (1/20) for 1 g of PCB respectively), thus raising the DES consumption. Ideally, a green solvent can extract as much metal with as little solvent as possible. Therefore, based on this optimization, an S/L ratio of 1/20 was selected for further study.

### 3.4. Ternary DES Synthesis

In the synthesis of the ternary DES, choline chloride served as the hydrogen bond acceptor (HBA), while glycerol and citric acid acted as the hydrogen bond donors (HBD). Choline chloride initially appeared as a white crystal, glycerol as a viscous liquid, and citric acid as a white solid. The three components were mixed in a molar ratio of choline chloride:glycerol: citric acid of 7:10:3, representing the eutectic point of this ternary DES as reported by Taysun *et al.* [24]. The molar ratio leads to a freezing point of 241 K for the ternary DES, which is lower than the freezing points of choline chloride (575 K), glycerol (291 K), and citric acid (429 K) [24, 26]. The synthesized ternary DES was placed in a desiccator and showed no appearance of solids or color changes after 60 days. The stability signifies the successful synthesis of ternary DES [26].

Table 2. Physical properties of ternary DES and its constituent components

Physical property	Choline chloride	Glycerol	Citric acid	Ternary DES
Appearance	Crystalline	Viscous liquid	Solid	Viscous liquid
Color	White	Colorless	White	Colorless
Stability	Hygroscopic	Stable	Stable	Stable
Density	1.02 g/mL	1.25 g/mL	1.67 g/mL	1.21 g/mL
pH	5-7.5	5.5-8	1.7	3

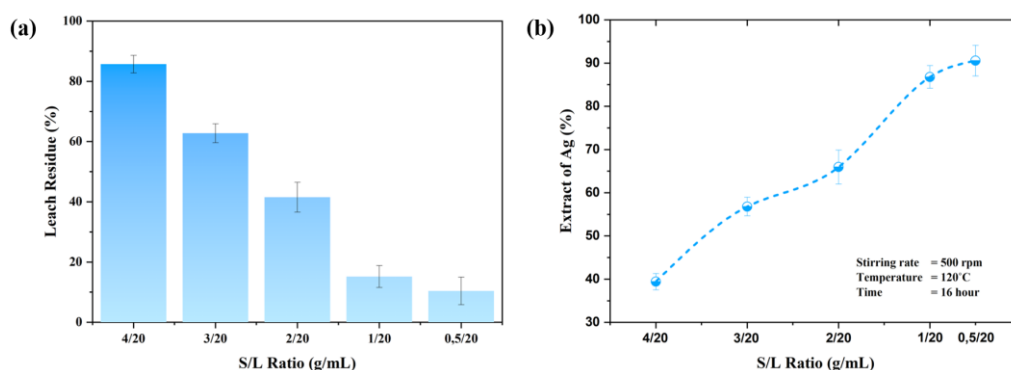


Figure 3. S/L ratio optimization: (a) the percentage of residue produced after leaching and (b) the percentage of extracted silver

### 3.5. Characterization of Ternary DES

The ternary DES was characterized by observing its physical properties, including appearance, color, stability, density, and pH, as detailed in Table 2. The ternary DES has a 1.21 g/mL density, higher than the glyceline DES. This discovery is in accordance with the research by Singh *et al.* [33], which reported increased hydrogen bond donor quantity in DES, leading to increased density. Moreover, the “hole” theory proposed by Abbott *et al.* [34] supports this explanation, suggesting that DES’s packing or molecular structure affects its density. The ternary DES has a pH of 3, which is lower than DES glycerine, which has a pH of 5. Higher acidity levels in deep eutectic solvents (DES) are critical for enhancing the dissolution of metal oxides during leaching processes, as the fundamental factor in dissolving metal oxides in DES is the activity of H<sup>+</sup> ions [35].

FTIR is an excellent method for characterizing samples by confirming functional groups or bonds within molecules [36]. The ternary DES and three constituents were analyzed using an FTIR spectrometer, as shown in Figure 4. The FTIR analysis results indicate that the choline chloride’s sp<sup>3</sup> C–H stretching peak appears at around 3000 cm<sup>-1</sup> [24]. C–H bond peaks are also evident in the DES spectrum, overlapping with the broad peaks of O–H alcohol from glycerol and the O–H carboxylic acid peaks from citric acid. Furthermore, the DES spectrum includes characteristic peaks of the C=O bond of carboxylic acid at approximately 1728 cm<sup>-1</sup> [24]. These peaks are present in the DES spectrum, strongly

indicating the intact structure of citric acid within the DES. All peaks in the spectra of choline chloride and glycerol are also observed in the spectrum of ternary DES, indicating that the HBD and HBA components maintain their chemical structures in the resulting DES. Details of the FTIR wavenumbers for the choline chloride-based (ChCl), glycerol (Gly), and citric acid (CA) ternary DES synthesis are listed in Table 3.

According to AlOmar *et al.* [26], Gly in DES exhibits O–H stretching in the 3200 to 3600 cm<sup>-1</sup> range, and for ChCl in DES, C–H stretching overlaps with O–H between 3000 and 3400 cm<sup>-1</sup>. Peaks around 1410–1485 cm<sup>-1</sup> indicate CH<sub>2</sub> bending, while CH<sub>3</sub> bending is seen around 1355–1400 cm<sup>-1</sup> [37]. Peaks around 1060–1100 cm<sup>-1</sup> indicate C–O bending, and there is an indication of N–C at around 950–960 cm<sup>-1</sup> [17]. The FTIR analysis results show that the ternary DES exhibits broadening and shifting of the O–H bond peaks compared to the spectra of Gly and CA.

The O–H peaks of Gly and CA were initially observed at approximately 3394.83 and 3367.82 cm<sup>-1</sup> and then shifted to 3385.18 cm<sup>-1</sup> in the ternary DES. This shift indicates the weakening of the O–H bonds. However, the citric acid peak shifts to a larger wavenumber in the ternary DES. This may be due to the presence of monohydrate in the citric acid, similar to the findings by Shafie *et al.* [38]. This shift is caused by more chloride ions (Cl<sup>-</sup>) in ChCl, which induce stronger hydrogen bond interactions with hydroxyl groups in monohydrated citric acid.

Table 3. Identification of ternary DES functional groups

Sample	Wavenumber (cm <sup>-1</sup> )					
	O–H stretching	C=O bond	CH <sub>2</sub> bending	CH <sub>3</sub> bending	C–O stretching	C–N stretching
ChCl	3362.04	-	1483.31	1399.61	1084.03	954.8
Gly	3394.83	-	1415.80	-	1041.60	-
CA	3367.82	1716.70	1419.66	-	1116.82	-
TDES	3385.18	1728.28	1471.74	1398.44	1051.24	956.72

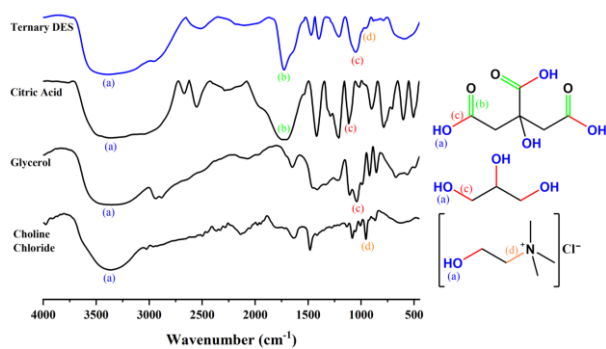


Figure 4. FTIR spectra of ternary DES and its constituents

A broad spectrum in citric acid and higher O–H peaks than in DES indicate a strong influence of monohydrate. This finding suggests that the ternary DES contains water, which will certainly affect the leaching of metals in PCB samples. Nevertheless, this does not affect hydrogen bonding, as explained by Hammond *et al.* [39], who stated that most DESs maintain their typical hydrogen bonding patterns even when the water content reaches 42 wt%. Therefore, the monohydrate content in citric acid ensures that the mixture retains the characteristic behavior of DES rather than becoming a dilute solution. The inclusion of water in DES for metal leaching has been used by Peeters *et al.* [23] in a choline chloride–citric acid DES with a water content of 35%, resulting in a leaching of 99.6% of  $\text{LiCoO}_2$ . Therefore, water in DES has shown positive results in metal leaching, as it can reduce the viscosity of DES without disrupting the typical hydrogen bonding patterns in the DES.

### 3.6. Leaching of Silver Using Ternary DES

The silver leaching process using DES is influenced by the proton activity ( $\text{H}^+$ ) and the strength of the hydrogen bond donor (HBD) [35, 40]. In this study, citric acid was added to glyceline binary DES to improve the efficiency of silver extraction. This modification is renowned for its high acidity due to multiple hydrogens ( $\text{H}^+$ ) ions, as reflected by its  $\text{pK}_a$  value of around 3.13 [41]. Therefore, forming ternary DES is anticipated to improve the strength of HBD and enhance the efficiency of silver metal extraction.

The silver extraction from PCB using ternary DES was carried out at the optimal conditions for leaching with glyceline DES. Following the leaching procedure, the samples were centrifuged to separate the undissolved residue. The XRF analysis data in Figure 6 indicate that silver extraction reached 93.65% under optimum conditions and provides a higher silver extraction yield compared to glyceline DES (86.77%). This outcome can be attributed to using citric acid as an HBD, which possesses a high acidity level, consequently enhancing proton activity to break metal's oxide bonds and accelerating the chemical reaction kinetics [22]. However, the citric acid presence also contributes to increasing the DES viscosity due to its relatively large molecular size [23]. On the other hand, the presence of glycerol as an HBD with smaller liquid molecules at room temperature improves the diffusivity of the DES. This condition enhances the mobility of molecules during the dissolution process [22].

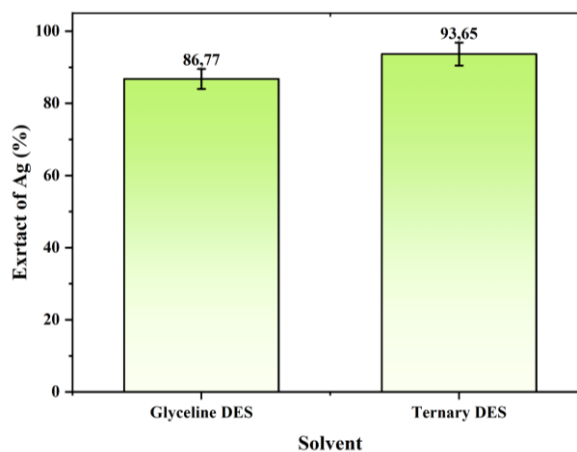


Figure 5. Percentage of silver metal extracted from PCB using glyceline and ternary DES

Ternary DES exhibits better potential in extracting silver metal from PCB waste samples. In addition, there is still room for further improvement in silver extraction using ternary DES, as the current leaching conditions are optimized for silver metal leaching using glyceline DES. Hence, optimizing leaching parameters using ternary DES in upcoming research is essential. These discoveries have important implications for developing more effective and eco-friendly solvents for recovering valuable metals from electronic waste.

## 4. Conclusion

The optimal silver metal leaching from PCB was achieved by utilizing glyceline DES at  $120^\circ\text{C}$  for 16 hours, with a 1/20 solid-to-liquid ratio, resulting in an efficiency of 86.77%. Ternary DES was successfully synthesized by adding citric acid to support HBD in glyceline DES. Ternary DES was viscous, colorless, and stable for 60 days, with lower acidity compared to binary DES with a density of 1.21 g/mL. FTIR analysis indicated the presence of hydrogen bonding in ternary DES, as evidenced by spectrum broadening and shifting. Ternary DES leached 93.65% of silver from PCB waste, outperforming glyceline DES with 86.77%. There remains potential to increase the silver leaching efficiency by optimizing leaching conditions using ternary DES, thus offering a sustainable option for electronic waste management.

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