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Solvometallurgical Extraction of Refractory Gold using Deep Eutectic Solvents

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Abstract – *The extraction of gold from refractory ores presents a significant challenge in the mining industry. This study investigates a novel approach of using deep eutectic solvents (DES) mixtures in extracting gold from refractory ores. Comparative leaching experiments were conducted on a refractory gold ore (P80 37 µm) at 30°C, employing choline chloride as the HBA and four HBDs: urea, malonic acid, ethylene glycol, and oxalic acid. The highest gold concentration (2.95 ppm) and recovery (48.9%) were achieved with the choline chloride-oxalic acid DES, with a clear trend of increasing gold extraction corresponding to increasing HBD acidity. The influence of temperature (30–60°C) and solids-to-lixiviant ratio (1:15 to 1:25) on gold extraction using the choline chloride-oxalic acid DES was then investigated. Results demonstrated that lower temperatures and higher lixiviant ratios significantly enhance gold extraction. These findings highlight the significant potential of DES to improve the efficiency and sustainability of refractory gold processing.*

Keywords*: deep eutectic solvents, refractory gold, choline chloride, gold extraction*

I. INTRODUCTION

The escalating global demand for gold, driven by its critical role in finance and technology, necessitates the development of more sustainable and cost-effective extraction methods. The depletion of readily accessible free-milling ores has forced a shift towards complex and refractory ores, which present significant challenges due to their mineralogical complexity and incompatibility to conventional extraction techniques. Gold from refractory ores is often difficult to liberate and recover due to the encapsulation of gold in the host mineral, possibly by physical factors (i.e. sulfides, oxides, and silicates), chemical factors (e.g. electrum and tellurides), substitution in the lattice (e.g. arsenopyrite), or by surface passivation resulting from a chemical layer formation [1, 2, 3, 4, 5, 6]. A refractory ore may also have the presence of carbonaceous matter which adsorbs the dissolved gold, a process known as preg-robbing, thus greatly reducing the extraction efficiency of the process [7]. Methods for the leaching of refractory gold ore include standard cyanidation and carbon adsorption processes. Depending on the mineralogy and degree of refractoriness, recovery from refractory gold ores typically is less than 80% (even up to 50%) [3]. Pretreatment is commonly applied to make such processes effective, such as roasting, bio-oxidation, and pressure oxidation [8, 9, 10].

The current processing techniques for extracting refractory gold are conducted through hydrometallurgical processes using aqueous media [11]; however, it has economic and environmental drawbacks. For example, gold cyanidation is widely adopted for leaching gold using cyanide because of its efficiency and cost-effectiveness, but its high degree of toxicity for human health and the environment are enough reasons to research alternative methods with comparable economic viability [2]. Hydrometallurgical methods also consume great amounts of acids and bases that generate aqueous wastes requiring additional treatment or neutralization before disposal [14]. On the other hand, pyrometallurgical techniques have also been explored to extract low grade refractory gold ores (e.g. selective chlorination process) [12]. Although pyrometallurgical methods can be effective, it is laborious and have a high energy demand since it operates at high temperatures (i.e. roasting and smelting in a furnace), and it releases sulfurous gasses which need to be captured and treated [13]. Looking at solvometallurgy, an emerging branch of extractive metallurgy, the problem of laborious processes, energy-intensive, low gold extraction, safety, and sustainability considerations may be answered.

Solvometallurgy uses non-aqueous solutions such as molecular organic solvents, ionic liquids, deep eutectic solvents (DES), or inorganic solvents (e.g. liquefied ammonia, concentrated sulfuric acid or supercritical carbon dioxide) to extract metals from ores [3]. Ionic liquids and deep eutectic solvents may also be considered as green solvents due to their non-volatility, low flammability, and low toxicity [4]. Both ILs and DES have comparable properties; however, ILs consist mainly of ions while DES may have neutral molecules which results in a lower melting point upon certain combinations [3]. This study focuses on the use of DES in treating refractory gold ore.

Deep Eutectic Solvents (DES) are eutectic mixtures of Lewis or Bronsted acids and bases containing various anionic and cationic species, classified as either hydrogen bond donors (HBDs) and acceptors (HBAs). DESs are composed of large, nonsymmetric ions with low lattice energy, and consequently, low melting points [14]. Further defining DES, eutectic mixtures are reversible isothermal non-reacting mixtures of different compounds formed during the cooling of a liquid system that result in a novel mixture with a lower freezing point compared to those of the pure compounds individually. The larger the interaction of the different compounds, the larger will be the temperature change, as shown schematically in Figure 1 [15].

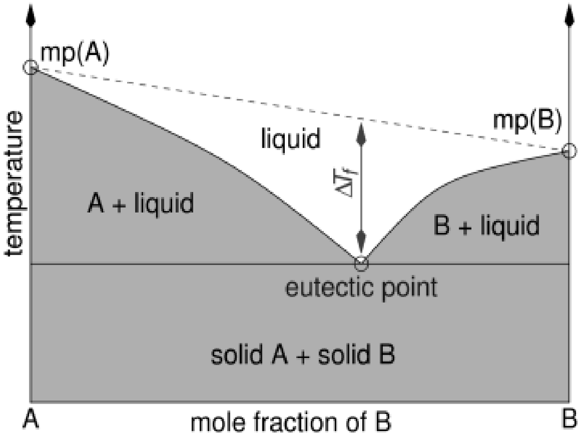

Figure 1. Eutectic point of a two-component phase diagram.

The study aims to investigate the use of Deep Eutectic Solvents (DES) mixtures in extracting gold from refractory ores. The research has the following specific objectives:

1. Explore the applicability of ChCl-based DES mixtures in leaching gold,
2. Determine the best ChCl-based DES in terms of gold recovery; and,
3. Investigate the effects of temperature and solids-to-liquids ratio on gold recovery of the best DES.

Driven by the need for more sustainable and viable extraction methods, this study employs deep eutectic solvents as the principal non-aqueous solvent. DES presents a compelling alternative due to their inherent potential for minimizing environmental and public health hazards, combined with the novelty of their application in refractory gold ore processing. Their wide compositional versatility and the availability of low-cost, readily accessible components, especially compared to ionic liquids, offer significant economic benefits. Additionally, DES contributes to decreased water and energy usage, aligning with key sustainability goals.

II. METHODOLOGY

The leaching experiment was done in two phases. First, the ore sample was leached using four mixtures with choline chloride (ChCl) as the HBA and urea (U), malonic acid (MA), ethylene glycol (EG), and oxalic acid (OA) as the HBDs. The mixture that obtained the highest gold recovery was then subjected to the second phase, wherein the leaching temperature and solids-to-lixiviant ratio were varied.

2.1 Ore Characterization

According to the mineral liberation analysis (MLA) data from Project 2 of the Green Tech Program, the ore is mainly comprised of pyrite (52.74wt%), quartz (28.36wt%), and chalcopyrite (5.83wt%), with a small amount of gold (0.00718wt%). The gold is predominantly present in the form of Au-Ag tellurides (77.27%), followed by electrum (12.57%) and native gold (8.87%), with about 80% of the grains measuring less than 12 microns. The occurrence of gold minerals is primarily associated with pyrite (66.22%) and pyrite-chalcopyrite-sphalerite (23%) and is typically locked on the sides of the minerals, although some free grains are also present (8.22%). These results confirm that the ore is a refractory gold ore.

2.2 Sample Preparation

The refractory gold ore was provided by a gold mining operation. The dry ore was mixed, homogenized, and split into representative samples for mineralogical characterization through the Mineral Liberation Analyzer (MLA), size analysis, and the leaching tests. The dry ore initially had an 80% passing particle size (P80) of 362 microns. The ore was further pulverized to P80 37 microns to lessen the effects of incomplete liberation and hindering the gold from being leached. A head grade of 10 ppm was determined using Atomic Absorption Spectroscopy (AAS).

2.2.1 Solvents used in this Study

Choline [cholinium cation or (2-hydroxyethyl)-trimethylammonium cation] was used due to its low toxicity and cost. It is classified as a provitamin and widely produced as an animal feed supplement [14]. As choline is mixed with HBD, high solubility of various metal oxides can be achieved. With a varying HBD reagent, the solubility of metal oxides will also vary to great degrees. The HBDs used in this study were urea, malonic acid, ethylene glycol, and oxalic acid. Urea has been one of the most commonly used HBD to partner with ChCl since it has a low cost of raw materials and a simple synthesis, is easy to biodegrade, and is non-toxic as a potential heavy metal eluent [16]. Malonic acid (maline) has been studied to have had high solubility of metal oxides, such as nickel, cobalt, and lanthanide oxides [17]. Ethylene glycol (antifreeze) is the most common HBD because of its desirable properties such as high boiling point and low vapor pressure. It is a thermally stable solvent, making it useful for leaching applications [3]. Oxalic acid is a weak acid. Using oxalic acid with choline chloride has found success in recovering bismuth and other metal oxides from blast furnace dust [18].

2.3 Comparative Leaching

The DES mixtures used for the leaching experiments were ChCl: urea, ChCl: malonic acid, ChCl: ethylene glycol, and ChCl: oxalic acid. The leaching experiments were conducted at a particle size of P80 37 microns. The setup involved a covered erlenmeyer flask while being agitated using a magnetic stirrer on top of a hot plate. Each run used five (5) grams of refractory gold concentrate to maintain a 1:15 solids-to-lixiviant ratio. A heating method was employed to achieve a homogenous DES mixture before adding the oxidant, iodine (0.1 M), subsequently followed by the ore. The temperature was kept constant at 30^{o}C and agitation at 300 RPM for 24 hours. The pH and ORP (oxidation-reduction potential) were measured before and after the leaching. After leaching, the mixture was filtered using a Whatman filter, and the filtrate was carefully stored in a vial, while the solids were set aside. The filtrate was subjected to X-ray fluorescence (XRF) to determine the gold concentration. Duplicates were done for the experiments. The design and conditions used for the comparative leaching are listed in Table 1.

Table 1. Testing conditions for the Comparative Leaching.

Run	**DES**	**Molar Ratio**	**Particle Size, microns**	**Oxidant, *M***	**Agitation, *RPM***	**Leach time, *hours***	**w/w ratio**
A	ChCl: Urea	1:2	37	0.1	300	24	1:15
B	ChCl: Malonic acid	1:1	37	0.1	300	24	1:15
C	ChCl: Ethylene glycol	1:2	37	0.1	300	24	1:15
D	ChCl: Oxalic acid	1:1	37	0.1	300	24	1:15

DES Selection

The recorded data were organized and evaluated. The goal of this phase was to compare and rank the DES mixtures in the order of most recommended lixiviant for leaching refractory gold ore in terms of its gold recovery. The DES with the highest recovery was determined and used for the investigation of leaching parameters.

2.4 pH and ORP levels

Throughout the leaching process, the pH and oxygen-reduction potential (ORP) were constantly measured using a pH - ORP meter. This was done to determine the tendency of the DES systems to lose or gain electrons through its hydrogen ions.

2.5 Investigation of Leaching Parameters

The effects of temperature and solids-to-lixiviant ratio were investigated using the highest-ranked DES mixture determined from the previous experiment. Table 2 shows the 2^2 factorial design of the experiment. Duplicates were also done for the experiment. The filtrates were measured for gold using the XRF. The data resulting from XRF were enough to observe the relationship of the parameters chosen, thus the employment of analytical measurement with higher accuracy was not deemed necessary. The equipment used provided an approximate detection limit for gold of 1 ppm (parts per million).

Table 2. Testing conditions for the Investigation of Leaching Parameters.

Run	**Oxidant, *M***	**Agitation, *RPM***	**Leach time, *hours***	**S:L ratio**	**Temp (ºC)**
1	0.1	300	24	1:15	30
2	0.1	300	24	1:15	30
3	0.1	300	24	1:15	60
4	0.1	300	24	1:15	60
5	0.1	300	24	1:25	30
6	0.1	300	24	1:25	30
7	0.1	300	24	1:25	60
8	0.1	300	24	1:25	60

III. RESULTS AND DISCUSSION

3.1 Refractory ore characterization

The refractory nature of the ore is confirmed by mineralogical analysis (MLA), which identified a dominant mineralogy of pyrite (52.7 wt%), quartz (28.4 wt%), and chalcopyrite (5.8 wt%), with a low gold grade (0.007 wt%). Gold occurs primarily as Au-Ag tellurides (77.3%), with subordinate electrum (12.6%) and native gold (8.9%). The fine dissemination of gold, with the majority of grains smaller than 12 µm, and its close association with pyrite (66.2%) and pyrite-chalcopyrite-sphalerite (23.0%) are key indicators of refractoriness.

3.2 Viability of using DES for Gold Extraction

The extraction of gold was investigated using different deep eutectic solvent (DES) mixtures based on choline chloride (ChCl). Gold concentrations and recoveries are detailed in Table 3, and average recoveries from duplicate trials are shown in Figure 2. Statistical analysis, employing t-tests to compare duplicate trials and ANOVA to assess the effect of DES composition, confirmed the reproducibility of the experiments and the significant influence of DES composition on gold recovery (see Appendix 1). Among the tested DES mixtures, only ChCl: urea exhibited a statistically significant difference in gold recovery compared to other groups, yielding the lowest recovery at 33.2%. Meanwhile ChCl: ethylene glycol, ChCl: malonic acid, and ChCl: oxalic acid achieved comparable recovery values at 43.4%, 46.4%, and 48.9%, respectively. Since ChCl: oxalic acid achieved the highest recovery, it was selected for subsequent investigation of leaching parameters.

Table 3. Leached Solution Concentration and Recoveries.

DES	**Concentration (ppm)**	**Recovery (%)**
ChCl: Urea	2.00	33.2
ChCl: Malonic acid	2.80	46.4
ChCl: Ethylene glycol	2.62	43.4
ChCl: Oxalic acid	2.95	48.9

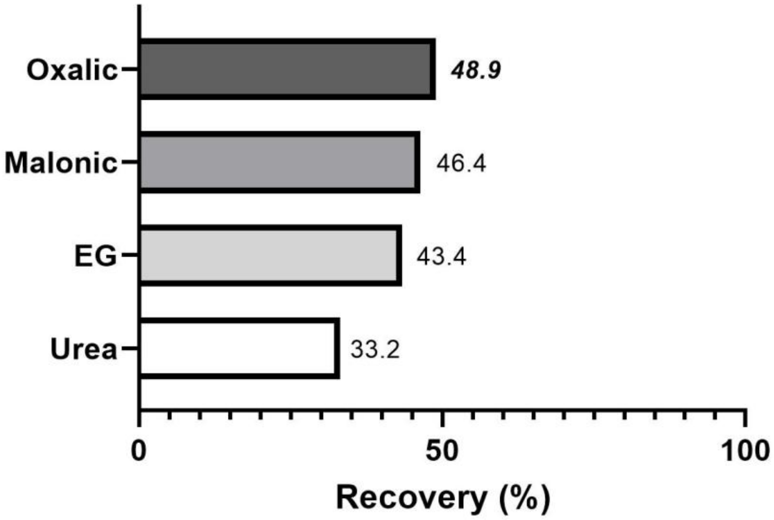

Figure 2. Recoveries of various ChCl-based DES mixtures.

Among the tested DES mixtures, ChCl: oxalic acid exhibited the highest recovery, followed in descending order by ChCl: malonic acid, ChCl: ethylene glycol, and ChCl: urea. Although all four DES mixtures demonstrated the capacity to extract gold from the refractory ore, their relative performance varied considerably, as discussed in the following subsections.

3.2.1 ChCl: Urea

The choline chloride-urea deep eutectic solvent (DES), commonly known as reline, is a tractable ionic liquid at room temperature. As shown in Table 3, this DES yielded an average gold concentration of 2.00 ppm, corresponding to a 33.2% recovery. The urea component, acting as a hydrogen bond donor, facilitates the dispersion and solubilization of certain ore components, potentially exposing trapped gold for extraction [20]. However, urea's weak hydrogen bonding with Au^{3+} through its oxygen atoms contributes to gold solubilization to a lesser extent compared to other choline chloride-based DES [21].

3.2.2 ChCl: Malonic Acid

The extraction of gold using the choline chloride-malonic acid system resulted in an average gold concentration of 2.80 ppm, corresponding to a 46.4% recovery. The mechanism of gold dissolution is attributed to the proton donation from malonic acid, a dicarboxylic acid, leading to the formation of Au^{3+}. This allows for the interaction of both carboxylic groups of malonic acid with the gold ion, potentially through both hydrogen bonding and chelation, thus promoting its dissolution [22]. Furthermore, the choline chloride-malonic acid DES exhibits superior thermal and chemical stability relative to other choline chloride-based DESs, suggesting its suitability for sustained gold leaching processes [23].

3.2.3 ChCl: Ethylene Glycol

The extraction of gold using the choline chloride-ethylene glycol system resulted in an average gold concentration of 2.62 ppm, corresponding to a 43.4% recovery. Ethylene glycol acts as a co-solvent enhancing the solubility of gold by lowering the overall viscosity and polarity of the solution [24]. This decrease in viscosity facilitates molecular mobility, thereby rendering the gold more accessible to extraction. While the potential for hydrogen bonding and redox reactions involving ethylene glycol exists, contributing to gold dissolution, these effects are less pronounced than those observed with oxalic acid [25].

3.2.4 ChCl: Oxalic Acid

The extraction of gold using the choline chloride-oxalic acid system resulted in an average gold concentration of 2.95 ppm, corresponding to a 48.9% recovery. The mechanism of gold dissolution involves proton donation from oxalic acid, leading to the formation of Au^{3+}, which enhances its solubility in the solution. The high relative recovery observed is attributed to the formation of stable gold complexes with oxalic acid [22]. Furthermore, the possibility of redox reactions, wherein oxalic acid acts as an electron donor, contributing to the dissolution process, should be considered [26].

Among the investigated hydrogen bond donors (HBDs), oxalic acid exhibited the highest efficacy for gold extraction from the refractory ore, based on the achieved recovery. While all four deep eutectic solvent (DES) mixtures demonstrated the potential for gold extraction, the ChCl:oxalic acid system achieved a maximum recovery of 48.9%, resulting in a gold concentration of 2.95 ppm. This enhanced performance can be attributed to the capacity of oxalic acid to form stable complexes with gold ions.

3.2.5 pH and ORP levels

The potential of hydrogen, or pH, was measured to measure the acidity or basicity of the system. The pH-ORP measurements, after the comparative leaching experiment, are shown in Table 4. The change in ORP measurements of the DES mixtures showed that the final oxidizing power decreased after leaching for ChCl with urea, ethylene glycol and oxalic acid, indicating that the consumption of the oxidizing species from the acid likely occurred during the leaching process. In contrast, an increase in the oxidizing power was observed for ChCl with malonic acid, indicating that the DES participated in a reduction while being consumed.

Although the increasing acidity (lowering pH level) is directly proportional to the increasing recovery of the used DES, the ORP data did not exhibit a similar relationship. The behavior of the ORP was not continuously monitored during the experiment, thus making it inconclusive to establish a trend with the recovery. Furthermore, the primary purpose of measuring ORP was not to correlate with gold dissolution but rather to provide insights into the possible mechanisms involved.

Table 4. Measured final pH and the change in ORP levels.

DES	Microns	pH (final)	ΔORP
ChCl: Urea	37	8.13	-220
ChCl: Malonic acid	37	0.48	+80
ChCl: Ethylene glycol	37	4.54	-230
ChCl: Oxalic acid	37	0.28	-270

Among the four DES combinations, only ChCl: urea showed to be a basic system. The highest gold extraction was achieved with ChCl: oxalic acid, the most acidic DES mixture. In addition to the previously discussed mechanisms, the enhanced formation of triiodide (I_3^-) at low pH (acidic conditions) likely contributed to gold extraction by facilitating the formation of gold-iodine complexes [27].

3.3 Investigation of Leaching Parameters

The choline chloride-oxalic acid mixture was selected for further investigation of leaching parameters due to its highest gold recovery among the four (4) DES combinations tested. The parameters under investigation were temperature and solids-to-lixiviant ratio. The temperature was varied from 30°C to 60°C, and the solids-to-lixiviant ratio was adjusted from 1:15 (66.7 g/L) to 1:25 (40.0 g/L). The resulting gold recoveries are illustrated in Figure 3. The most favorable extraction conditions were observed at lower temperatures and increased lixiviant ratios. A discussion of the potential mechanisms underlying the observed effects of each parameter is provided in the subsequent sections.

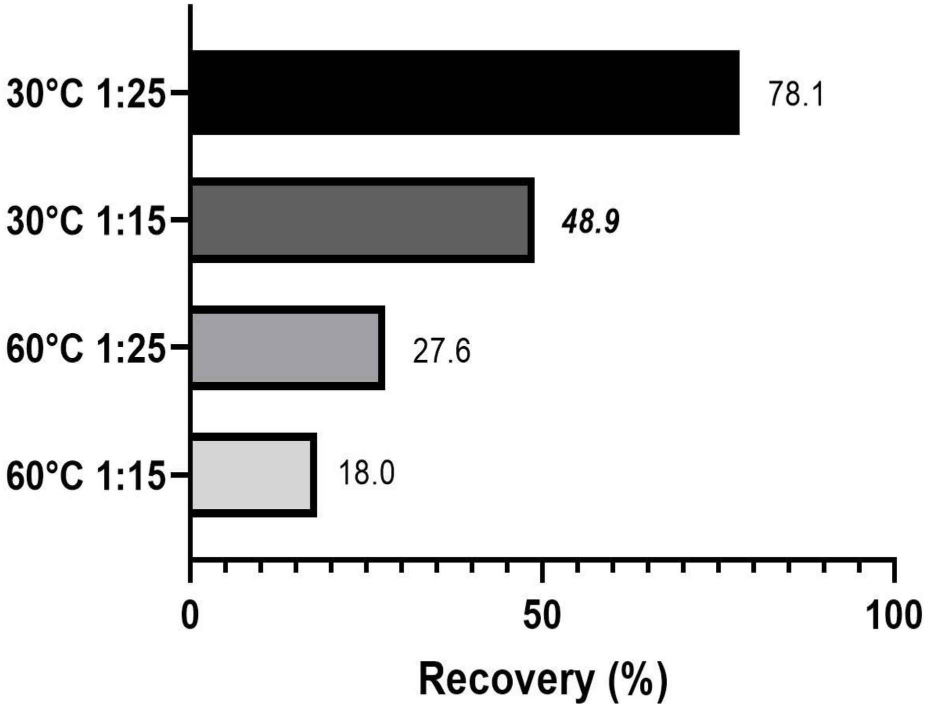

Figure 3. Gold recovery under varied leaching conditions.

3.3.1 Effects of Temperature

The resulting gold recoveries obtained under varying leaching conditions are presented in Figure 3. The highest gold extraction efficiencies were observed at lower temperatures and higher solids-to-lixiviant ratios. A discussion of the potential mechanisms contributing to the observed effects of each parameter is provided in the subsequent sections.

There are two potential reasons for this behavior. First, while choline chloride-oxalic acid itself is thermally stable [30], its interaction with other chemicals may not be. After filtering the solids, the observed gold concentration in the filtrate decreased when leached at a higher temperature. The amount of the collected crystals was inadequate for a precipitation analysis. Precipitates were formed when both lixiviants amount and temperature were increased as shown in Figure 4.

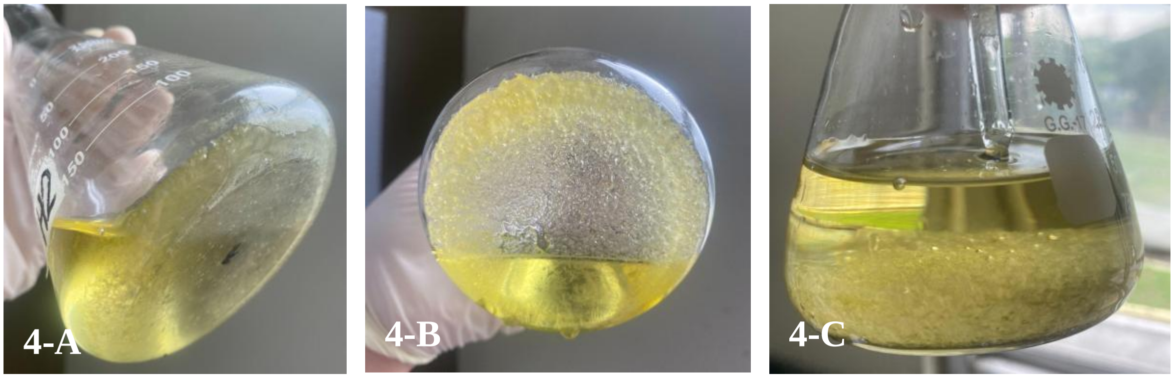

Figure 4. Precipitates in setups in varying temperatures and solids-to-lixiviant ratio; A: (-) Temp (+) S:L B: (+) Temp (-) S:L, and C: (+) Temp (+) S:L.

The precipitates formed in setups 4-B and 4-C exhibited greater solidification, which corresponded to reduced gold concentrations in the corresponding filtrates. In contrast, setup 4-A, performed at a lower temperature, resulted in the highest gold recovery. The precipitate formed in this setup was gelatinous and readily re-dissolved at room temperature, suggesting the potential for gold recovery from these precipitates over time. These observations demonstrate that temperature influences not only the formation of precipitates but also their physical characteristics, specifically their degree of solidification, which in turn significantly impacts the observed gold content in the liquid phase. The increased volume of crystalline residue observed in higher-temperature setups compared to room-temperature setups (Figure 5) further supports the hypothesis of reduced mixture stability at elevated temperatures.

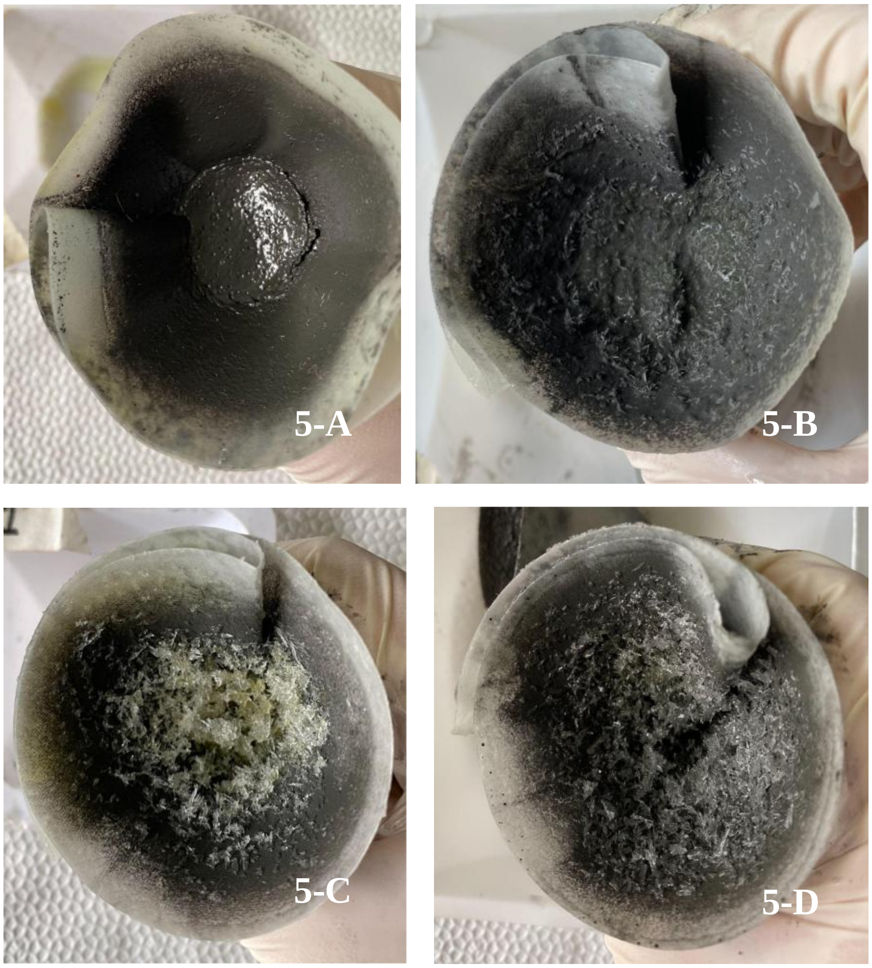

Figure 5. Residues of the setups and crystallization in varying temperatures and solids-to-lixiviant ratio; A: (-) Temp (-) S:L B: (-) Temp (+) S:L C: (+) Temp (-) S:L, and D: (+) Temp (+) S:L.

Another potential reason can be attributed to the competitive reaction of the lixiviant with other metallic species present in the ore matrix. Increasing the temperature can drive kinetics for other chemical reactions in the system to occur. Iron, as a component of pyrite, was a major component of the ore and was significantly present in much higher concentrations than gold. It was found that the concentration of iron also varied so it was potentially dissolved and affected by a change in the leaching conditions. When the available lixiviant reacts with iron, the probability for the lixiviant to react with gold would decrease, resulting in a decreased gold recovery. This suggests that the presence of other metals in the ore can consume the reagent intended for gold, potentially affecting its recovery.

Table 5. Iron concentration

Parameters		Iron Concentration (ppm)
S:L ratio	Temp (ºC)	
1:15	30	1965.33
1:15	60	797.25
1:25	30	371.75
1:25	60	174.50

3.3.2 Effects of solids-to-lixiviant ratio

The effect of solids-to-lixiviant ratio (1:15 and 1:25) on gold recovery using the choline chloride-oxalic acid DES was investigated. As shown in Figure 6, increasing the lixiviant ratio enhanced gold recovery at both temperatures. This is attributed to the increased availability of lixiviant, facilitating access to gold and promoting extraction [31].

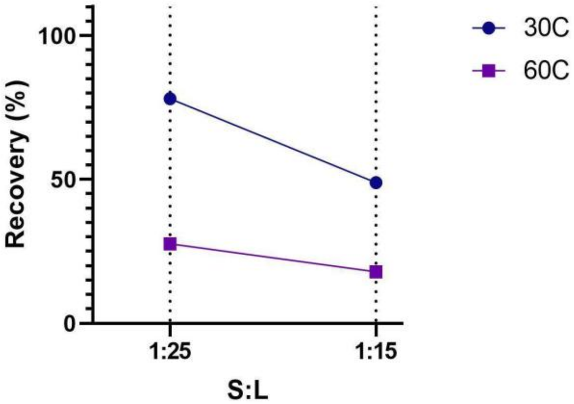

Figure 6. Effect of solids-to-lixiviant ratio on gold recovery.

Although elevated temperatures can increase leaching activity by reducing viscosity and enhancing reaction kinetics, the temperature sensitivity of the DES may also promote undesirable side reactions. In this study, increasing the temperature led to a decrease in gold recovery, suggesting the dominance of these competing processes. The observed increase in gold recovery with increasing lixiviant ratio is consistent with established literature, as more lixiviant becomes available to interact with gold.

This study found that DES is a promising lixiviant for the extraction of gold from refractory ores. However, its application for industry scale is still far from being potential. Significant challenges persist regarding the behavior and efficacy of DES, particularly due to limitations related to cost and the sensitivity of DES mixtures during synthesis and usage which can adversely impact the extraction process. Further studies should investigate the effect of leaching time on the recovery of gold under DES systems to optimize these parameters.

IV. CONCLUSION

The study investigated the solvometallurgical extraction of refractory gold using various deep eutectic solvent mixtures, examining the effect of temperature and solids-to-lixiviant ratio. Comparative leaching experiments demonstrated that all tested hydrogen bond donors (HBDs)—urea, malonic acid, ethylene glycol, and oxalic acid—combined with choline chloride as the hydrogen bond acceptor (HBA), were capable of extracting gold. The increasing acidity of the HBDs showed an increasing ability to leach gold. The choline chloride-oxalic acid DES yielded the highest gold concentration (2.95ppm) and recovery (48.9%). Subsequent investigation of leaching parameters using this DES revealed that increasing temperature decreased gold extraction, while increasing the solids-to-lixiviant ratio enhanced it. The highest gold recovery was obtained at 30°C and with a 1:25 solids-to-lixiviant ratio. While elevated temperatures can reduce viscosity and enhance ion mobility, they also promoted competing reactions and affected DES stability, thus negatively impacting extraction. Conversely, increasing the lixiviant ratio provided greater availability of lixiviant for gold interaction, leading to improved recovery. This study demonstrates the potential of DES as lixiviants for refractory gold extraction. However, challenges related to cost, synthesis, and sensitivity to operational conditions remain. Further optimization studies, particularly focusing on leaching time, are recommended to improve the efficiency and feasibility of DES for industrial-scale refractory gold extraction.

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