

Leaching of Refractory Gold Telluride Ore using Choline Chloride-Ethylene Glycol

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ABSTRACT: Processing refractory gold telluride ores presents a major challenge in conventional cyanidation because gold occurs as particles locked within sulfides, restricting contact with the lixiviant, and as gold tellurides that are insoluble in cyanide. To address this, alternative lixiviants such as deep eutectic solvents (DES) are being explored as gold ores become increasingly complex. This study investigates the leaching behavior of refractory gold telluride ore using ethaline or choline chloride-ethylene glycol, a type of DES, prepared at a 1:2 molar ratio. Leaching experiments were conducted at varying times (24, 48, and 72 hours), temperatures (30 °C and 55 °C), and particle sizes (37 µm and 74 µm), with a constant lixiviant-to-solid ratio of 1:10, 0.1 M iodine as oxidant, and agitation at 300 RPM. Gold concentration in the leachate was analyzed using Atomic Absorption Spectroscopy (AAS) to determine the percentage of gold dissolution. Results indicate that temperature had minimal effect on gold dissolution, suggesting that leaching can be effectively performed at ambient temperature. However, particle size and leaching time significantly influenced dissolution, with higher gold dissolution observed for smaller particles. Gold dissolution increased over time, reaching a maximum of 57% at 48 hours, followed by a decline, emphasizing the need to optimize leaching duration. Overall, results demonstrate that choline chloride-ethylene glycol is able to leach refractory gold telluride ores and may be an alternative to conventional cyanidation method.

KEYWORDS: Refractory Gold, Gold Telluride, Ethaline, DES

I. INTRODUCTION

The quality of readily available gold ores has become increasingly complex, leading to challenges in ensuring that global gold supply meets rising demand through conventional cyanidation methods. One of the most common available gold ore nowadays is refractory gold in which gold is physically locked within sulfide minerals primarily pyrite (FeS₂). This encapsulation hinders the direct accessibility of lixiviants to dissolve gold during leaching [1, 2, 3]. To enhance gold recovery, sulfide minerals such as pyrite must first be decomposed through a pretreatment process commonly using oxidation to improve gold recovery [4, 5].

Another challenge in gold extraction is that gold can be chemically associated with other elements such as tellurium forming gold tellurides [6]. Gold tellurides can be a source not only of gold but also tellurium that is used in electronics, renewable-energy technologies (such as solar panels), and other advanced materials [7]. However, gold tellurides cannot be dissolved by conventional cyanidation since it forms a passive layer of insoluble H₂TeO₃ layer which prevents cyanide from leaching gold [8].

To overcome the limitations of conventional cyanidation, alternative processes such as the use of deep eutectic solvents (DES) are being explored. DES are a new class of solvents formed by combining two solid components,

a hydrogen bond acceptor (HBA) and a hydrogen bond donor (HBD), creating a eutectic mixture at a specific ratio with a melting point significantly lower than that of the individual components. This results to liquid phase at relatively low temperatures. DES are biodegradable, non-volatile, and water-free. Thus, DES reduce the environmental risk of gold leaching [9, 10].

A commonly studied deep eutectic solvent is ethaline, which is composed of choline chloride (ChCl) with chemical formula of C₅H₁₄ClNO as the hydrogen bond acceptor (HBA) and ethylene glycol (EG) with chemical formula C₂H₆O₂ as the hydrogen bond donor (HBD), typically in a molar ratio of 1:2. Ethaline is notable for its thermal stability, low toxicity, and strong hydrogen-bonding capability, which enhances the dissolution of metal complexes, including gold. Its ionic nature also promotes the stabilization of metal ions in solution, providing an effective medium for metal recovery reactions. Its ionic and hydrogen-bonding network provides a favorable environment for stabilizing gold complexes, potentially overcoming the limitations of conventional cyanidation [11, 12, 13]. However, its application to refractory gold ores remains largely unexplored, presenting an opportunity for innovation in the field of green hydrometallurgy.

This study aims to investigate the leaching of refractory gold telluride ore using ethaline. The developed process seeks to provide an alternative to traditional cyanidation. Since ethaline is biodegradable and water-free, it can be safely handled while minimizing environmental impact and water resource consumption.

II. METHODOLOGY

A. Ore Preparation

The gold ore used in this study contains 18.20 ppm Au that occurs as 72.3% Au (Ag) telluride (selenide), 12.6% electrum, 8.9% native gold, and 1.3% Ag (Au,Bi) determined using the Mineral Liberation Analyzer (MLA). Major minerals present are pyrite (52.7 wt%), quartz (28.4 wt%), and chalcopyrite (5.8 wt%). Native gold is 92% associated with pyrite, with only 8% occurring as liberated. The Au (Ag) telluride (selenide), electrum, native gold, and Ag (Au,Bi) exhibit strong associations with pyrite at 70%, 62%, 92% and 49%, respectively showing that gold is indeed refractory. Eighty percent (80%) of the gold is smaller than 20 microns, indicating that gold predominantly exists as very fine particles within the ore.

The ore was dried, homogenized, and subjected to grinding to attain the desired particle size

B. Ethaline Preparation

Ethaline was prepared by mixing choline chloride (HBA) and ethylene glycol (HBD) at 1:2 mol ratio in an Erlenmeyer flask at 50°C and 300 RPM for 5-10 minutes. Once the mixture has become liquid, 0.1 M iodine was added. Iodine serves as an oxidizing agent ($E_{O_2/I_2/I^-} = 0.54V$), to initiate sulfide oxidation and facilitate the dissolution of gold [4, 14]. It was used since it is soluble in ethaline unlike chlorine and does not decolorize over 25 hours unlike bromine [15].

C. Determination of effects of time

After homogenizing the ethaline for 5 minutes, five (5) g of ore was added to attain a 1:15 solid to lixiviant ratio. According to Khuduwe et al. (2024), optimum solid to ethaline ratio is 3:10 [16]. However, 1:15 was used in this study to decrease solution viscosity and ensure that mass transfer is not the rate-limiting step.

Leaching was done at varying times following the parameters in Table 1 and setup shown in Figure 1 at ambient temperature of around 30 degrees Celsius. Constant agitation of 300 RPM was used following the study conducted by Bidari et al. (2024) [5]. Particle size of 37 microns was used since majority of the gold grains are in the finer particle size. Thus, leaching at this particle size ensures that particle liberation is not the rate-controlling step. The mixture was filtered after leaching, and the leachate was subjected to Atomic Absorption Spectroscopy (AAS) for gold determination. Duplicates were done per setup.

Table 1. Setup for Determining Effect of Leaching Time

Setup	Time, hrs
A	24
B	48
C	72

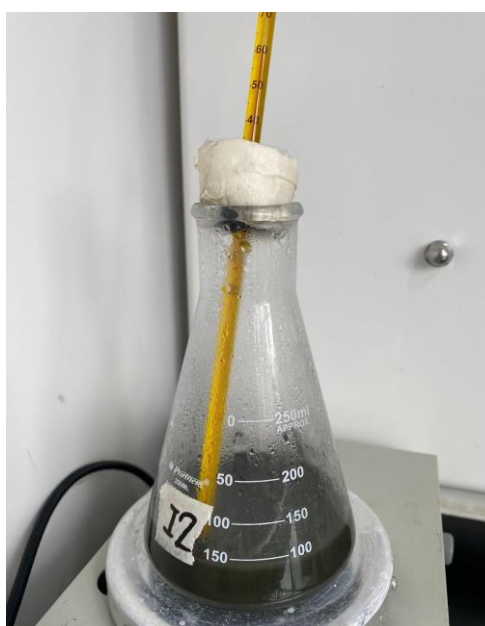


Figure 1. Leaching Setup

D. Determination of effects of particle size and temperature
Same setup as Section II.C was used but leaching was done at varying temperatures and particle sizes shown in

Table 1. Leaching was done for 48 hours since this duration yielded the maximum gold dissolution in Section II.C.

Table 2. Setup for Determining Effects of Particle Size and Temperature

Setup	Temperature, deg C	Particle Size, microns
D	30	37
E	30	74
F	55	37
G	55	74

E. Inferring Possible Mechanism

Same setup as Section II.D was used but leaching was extended to 120 hours. Solution pH, ORP, and aliquots were taken at certain intervals to explore the leaching behavior at extended times. The solution ORP was measured to assess its redox capability. Positive ORP indicates an oxidizing condition, while negative ORP indicates a reducing one. For sulfide oxidation and gold dissolution, the ORP must meet or exceed the respective reaction potentials. Samples before and after leaching were also subjected to Fourier-transform infrared (FTIR) spectroscopy to evaluate the possible interaction of ethaline with the ore.

significant effect on dissolution of gold at 95% confidence level which agrees with the observation by Khuduwe et al. (2024) in using ethaline to dissolve gold [16]. As shown in Figure 2, leaching exhibited an increase in gold dissolution over time, with the highest dissolution observed at 48 hours. Extended leaching duration allows more time for the lixiviant to interact with the ore and facilitate gold dissolution. However, beyond the 48th hour, a decline in gold dissolution was observed. The decrease in gold dissolution over time has also been observed by Bidari et al. (2024) when using ethaline with iodine in dissolving gold which could have been caused by precipitation of gold iodide. This phenomenon is likely due to the instability of the gold complex at lower solution potential, which can lead to precipitation under certain conditions [4].

III. RESULTS AND DISCUSSION

A. Effect of Leaching Time

Dissolution of gold behaves differently at different leaching time. Analysis of variance shows that leaching time has a

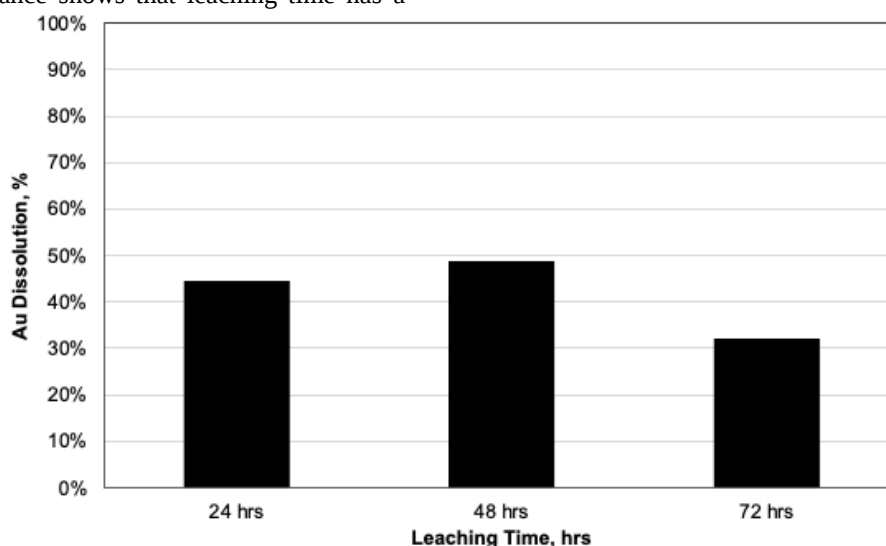


Figure 2. Gold dissolution (%) at varying leaching time

The stability of the gold complex is influenced by the solution’s pH and ORP (oxidation-reduction potential), both of which can be altered as the ethaline is consumed during leaching over time. These changes in the chemical environment can destabilize the gold complex, resulting in its conversion back to the solid phase. Therefore, excessively long leaching times are not desirable when using ethaline, as continuous consumption decreases solution potential creating

unfavorable conditions for maintaining gold in solution. However, Pourbaix diagram for gold in ethaline, which is currently not available, is still needed to be established to evaluate the solubility and stability of gold complex.

B. Effect of Temperature and Particle Size

Based on Figure 3, gold dissolution at leaching temperatures of 30 and 55 degrees Celsius are comparable

and not significantly different. Analysis of variance confirms that leaching temperature has no significant effect on gold dissolution at 95% confidence level which agrees with the observation from a study conducted by Khuduwe et al. (2024) [16]. Although increasing the temperature should provide more energy to the system to dissolve gold and should increase the rate of gold dissolution, it also hastens the consumption of ethaline. This lowers the solution potential which results in a solution condition of gold complex instability. This suggests that gold dissolution using ethaline can already be conducted at ambient temperature which would also be beneficial since no heating is required during leaching, thus using less energy.

Although temperature does not significantly affect gold dissolution, particle size does. Analysis of variance confirms that particle size has a statistically significant impact on gold dissolution. Gold dissolution increases as particle size decreases. This can be attributed to the higher surface area-to-volume ratio of finer particles, which provides more contact area between the particles and the lixiviant, thereby enhancing gold dissolution. Additionally, reducing particle size improves the liberation of gold, since gold is finely disseminated within the sulfide matrix. More liberated gold becomes available and accessible for reaction with the lixiviant.

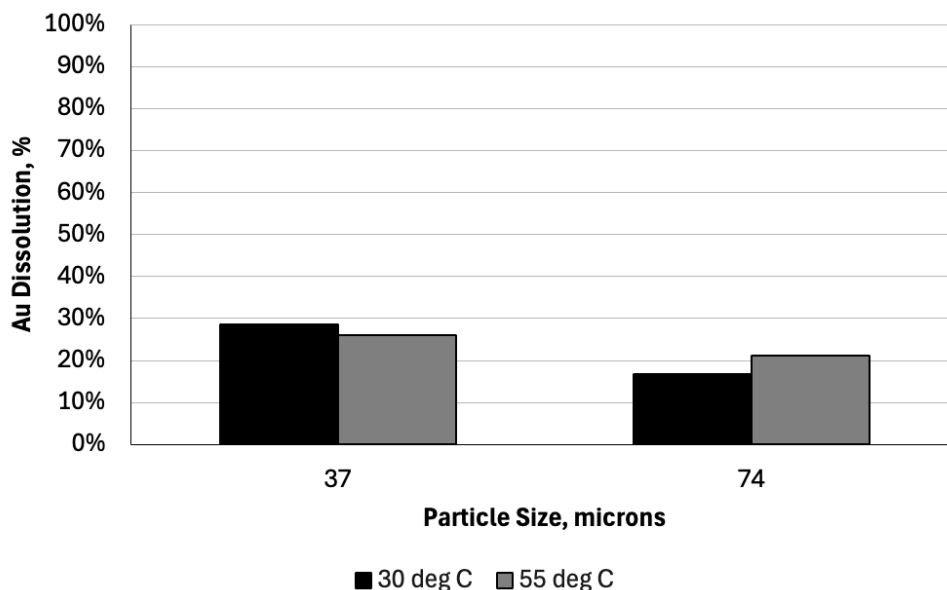
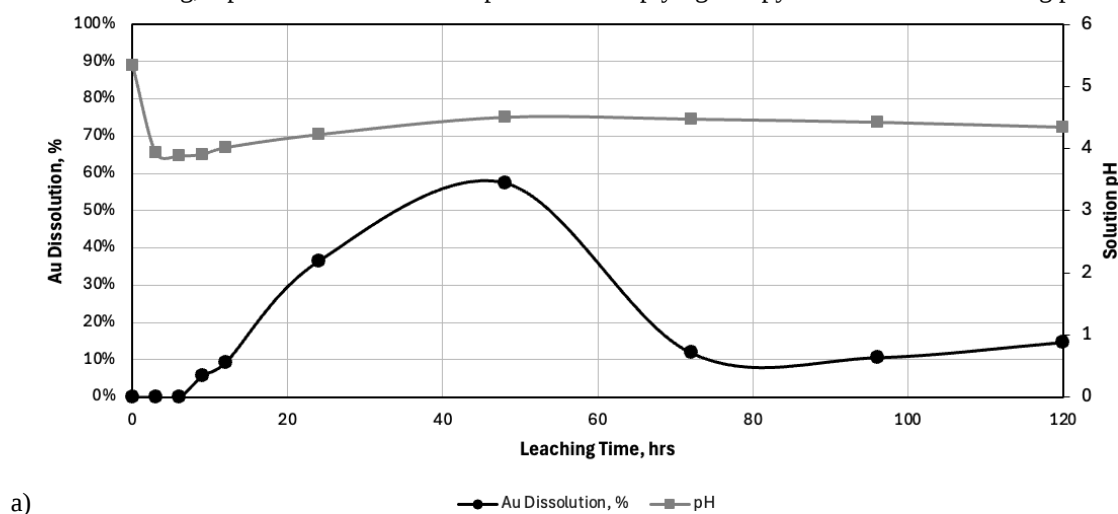


Figure 3. Gold dissolution (%) at varying particle size and leaching temperature

C. Possible Mechanism

It was observed that gold dissolution in ethaline proceeded relatively slowly, with noticeable dissolution occurring around the 9th hour of leaching, as shown in Figure 4a. Despite gold dissolution only becoming noticeable around the 9th hour of leaching, rapid decrease in solution pH

was still observed indicating that a certain reaction other than gold dissolution was taking place. Since the ore is mostly composed of pyrite, ethaline could have interacted with pyrite at the start of leaching. As indicated in Figure 4b, the solution potential exceeded the reaction potential for pyrite oxidation, implying that pyrite oxidation was taking place.



a)

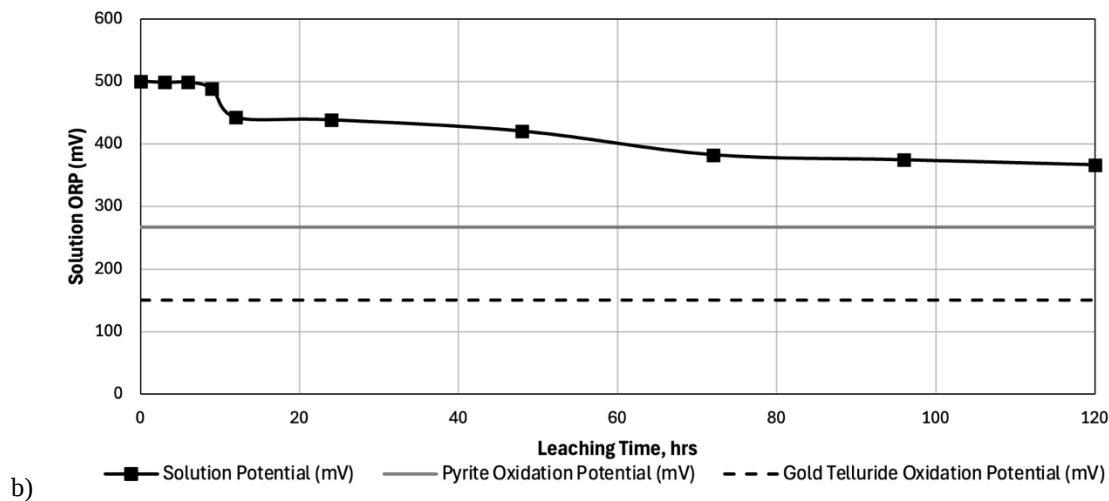
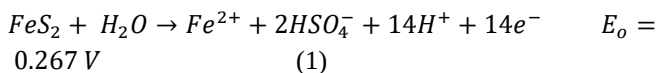
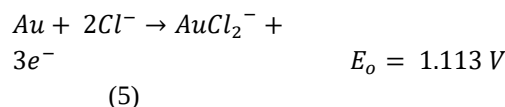
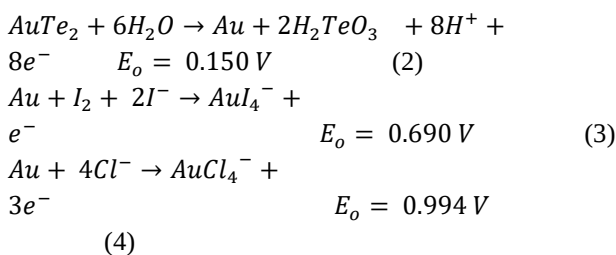


Figure 4. Gold dissolution with time a) %Au Dissolution and pH b) Solution ORP (mV)

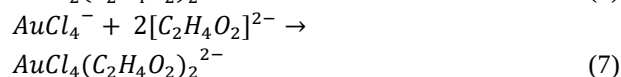
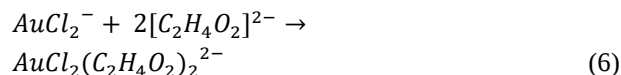
The drop in pH at the initial stage of leaching is therefore likely attributed to pyrite oxidation, as described by Equation 1, which generates H^+ ions and lowers the solution pH [4]. Even though ethaline is consumed during sulfide oxidation, the solution ORP was sustained possibly due to the release of H^+ ions.



The oxidation releases the encapsulated gold telluride and allowing it to be accessible by the ethaline. The exposed gold telluride reacts with the ethaline and oxidizes forming gold metal at pH 4 following Equation 2 [17]. As indicated in Figure 4b, the solution potential exceeded the reaction potential for gold telluride oxidation, implying that gold telluride oxidation was taking place. Once gold has been released from gold telluride, it can react with iodide from iodine and the chloride ions from choline chloride ($C_5H_{14}ClNO$) forming gold complexes represented by Equation 3, 4 and 5. However the measured solution potential shown in Figure 4b is less than the reaction potential of the formation of gold iodide and chloride complexes indicated in Equation 3, 4, and 5. This shows that gold iodide and chloride complexes are not stable during the leaching process using ethaline, and gold may have reacted with other anions in the system forming more stable gold complex.



Ethylene glycol can deprotonate forming $C_2H_4O_2^{2-}$ which can react with the gold chloride complex and may form gold complexes following Equation 6 and 7. Based on Figure 4, the production of $C_2H_4O_2^{2-}$ which is a conjugate base is evident as gold is dissolved since pH started to increase with gold dissolution. Based on the study by Faigal et. al that used density functional theory (DFT) to predict possible gold complex in ethaline leaching, $AuCl_2(C_2H_4O_2)_2^{2-}$ is the most probable gold complex that is stable in gold leaching using ethaline [18].



Based on the FTIR spectra in Figure 5, the O-H stretching in the ethaline with iodine slightly shifted from 3294 cm^{-1} to 3289 cm^{-1} after leaching indicating interaction of the solvent with the ore. The C-O stretching in ethaline at 1134 cm^{-1} is also no longer prominent after leaching. The shift in the O-H stretching and non-prominence of the C-O stretching vibration could have been caused by the deprotonation of ethylene glycol forming $C_2H_4O_2^{2-}$ which can react with the gold chloride complex. This confirms that $C_2H_4O_2^{2-}$ can act as a ligand to make a complex in ethaline as observed also by Teimouri et al. (2024) in leaching pyrite using ethaline [3].

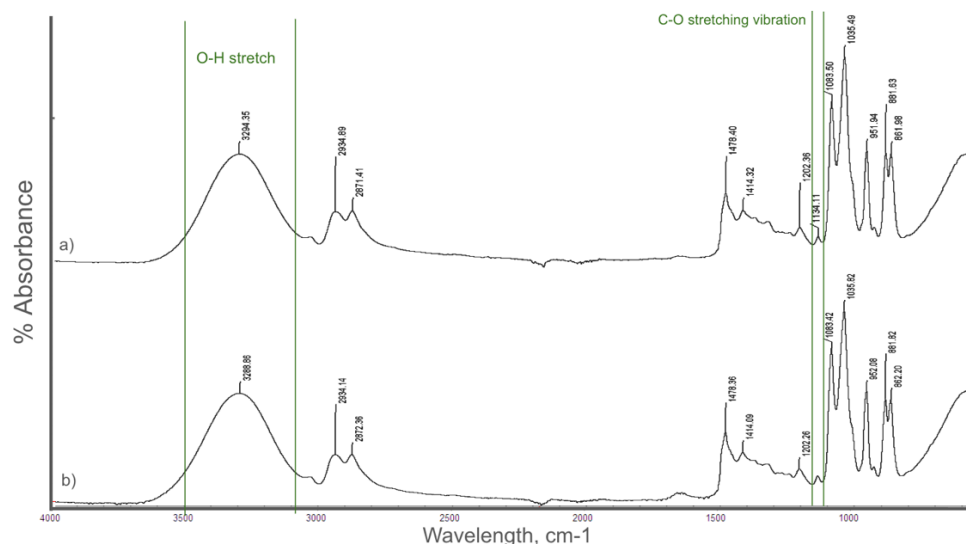


Figure 5. FTIR Spectra for a) Ethaline with 0.1 M Iodine b) Leachate

Gold dissolution exhibited an almost linear increase up to the 48th hour with continuous increase in pH and decrease in solution potential indicating continuous consumption of ethaline during leaching. This linear trend can be attributed to the initial concentration of the ethaline, during which a greater amount of active lixiviant is available to interact with the ore, facilitating consistent gold extraction. Beyond the 48th hour, the dissolution rate began to decline. This behavior suggests that a significant portion of the ethaline had been consumed, thereby reducing the availability of reactive species necessary for continued leaching. Prolonging the leaching time beyond 48 hours led to a reduction in gold dissolution, suggesting that extended leaching using ethaline may not be beneficial. This decline in performance is likely due to the instability of the gold complexes at low solution potential as indicated in the decrease of solution ORP. These findings highlight the importance of optimizing leaching duration to maximize gold dissolution.

Based on the results, ethaline can be a potential lixiviant for refractory gold telluride ore as it was able to dissolve significant amount of gold. However, further optimization is required to enhance the percentage of gold dissolution.

IV. CONCLUSION

This study investigated the leaching of refractory gold telluride ore using ethaline, a type of DES, prepared at a 1:2 choline chloride–ethylene glycol molar ratio. It was determined that gold dissolution is not significantly affected by temperature which indicates that leaching can be done at ambient temperature. Furthermore, particle size and leaching time have significant effect on gold dissolution. Increased gold dissolution is attained at smaller particle sizes. Leaching exhibited an increase in gold dissolution over time, with the highest gold dissolution of 57% observed at 48 hours. However beyond the 48-hour mark, a decline in gold dissolution was observed which highlights the importance of

optimizing leaching duration to maximize gold dissolution. Overall, the results show that choline chloride-ethylene glycol is able to leach refractory gold ore tellurides and can be further explored to determine optimum leaching parameters as alternative for conventional cyanide leaching.

V. ACKNOWLEDGEMENT

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