

Novel Extraction Process for Gold Recovery from Thiosulfate Solution Using Phosphonium Ionic Liquids

Harshit Mahandra,* Fariborz Faraji, and Ahmad Ghahreman*

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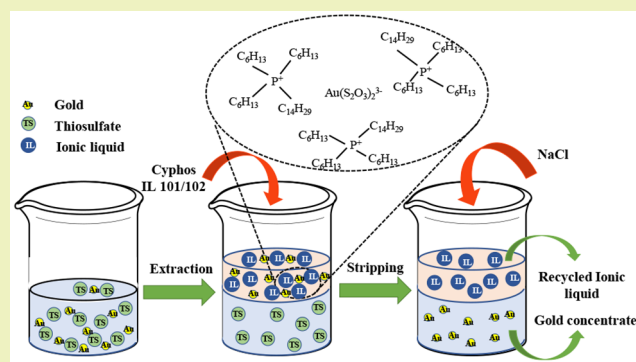
Article Recommendations



Supporting Information

ABSTRACT: A novel and sustainable technology to recover gold from thiosulfate medium using ionic liquids, i.e., Cyphos IL 101 (Cy IL 101) and Cyphos IL 102 (Cy IL 102) diluted in toluene, has been developed. Gold was extracted into the ionic liquid phase as $[\{P_{66614}^+\}_3\{Au(S_2O_3)_2^{3-}\}]$ and stripped using NaCl solution. The recyclability of ionic liquids has shown promising recirculation of the solvents for the extraction of gold from thiosulfate medium. Gold was quantitatively extracted from 0.2 mol/L sodium thiosulfate initially containing 10 mg/L gold at pH = 9.0 with 1.25 mmol/L ionic liquid (Cy IL 101 or Cy IL 102) in one stage at A/O = 2, whereas total gold stripping with 1.5 mol/L NaCl also needed one stage at A/O = 1. Using a high A/O ratio in the extraction stage (A/O = 10) and low (A/O = 1/10) in the stripping stage confirmed the economic and environmental viability of the process. The results revealed that Cy IL 101 presents slightly better behavior toward gold recovery than Cy IL 102 and is a viable and promising alternative to recover gold from the thiosulfate medium on a pilot scale. The overall study confirmed the suitability of the developed scheme for industrial application to provide economic and environmental benefits.

KEYWORDS: Extraction, Cyphos IL 101, Cyphos IL 102, Gold, Stripping



INTRODUCTION

Gold, a noble and precious metal has been extensively used in jewelry, electronics, aerospace, dentistry, orthopedics, etc.¹ The conventional gold extraction process from primary and secondary resources applies sodium cyanide as the most promising gold leaching lixiviant. The extreme toxicity of cyanide results in harmful environmental impacts.² Recently, the consumption of cyanide in gold processing has become prohibited in many regions.³ This has created an opportunity for the other viable lixiviant such as chloride, thiocyanate, thiourea, and thiosulfate to replace cyanide in gold leaching systems.² The use of thiosulfate as a lixiviant for gold leaching is widely accepted and is the most promising method because of its high selectivity, low cost, reduced environmental risk, and low corrosivity.^{2,4} In addition, thiosulfate leaching with carbonaceous ores has the advantage of avoiding the preg-robbing phenomenon (i.e., adsorption of the leached gold on the carbonaceous matter present in the ore which results in loss of gold to the tailings of the process and low efficiency of gold extraction process) during leaching which is the drawback with the cyanidation process. This is because gold thiosulfate complex has very low affinity for carbonaceous substances.⁵

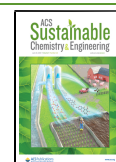
Although thiosulfate is the most promising alternative for cyanide in gold production, the scale up of thiosulfate leaching process for commercialization is still very slow and only Barrick

Gold Corporation in its Goldstrike mine (Elko, NV, USA) is employing the process.^{6,7} A major problem that limits the application of thiosulfate for gold recovery is the purification of the leach solution and gold recovery step. Considering that the thiosulfate complex does not effectively adsorb over activated carbon (which is used to selectively separate gold after or during the cyanidation process), the conventional processes such as carbon in pulp (CIP) operation are not applicable.⁸ This is due to the steric effects, highly negative charged complex, and complexing ligand-active carbon sites interactions.⁸ Therefore, alternative purification techniques including cementation,⁹ ion-exchange resin adsorption,¹⁰ mesoporous silica adsorption,¹¹ electrowinning,¹² and solvent extraction (SX)¹³ can be the preferred techniques to recover gold from thiosulfate solutions. However, cementation suffers from high reagent consumption, low purity of recovered gold, and hindrance in cyclic utilization of the thiosulfate solution.⁸ The loading of unnecessary metals and the elution of gold from

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resins is problematic in the resin-based ion exchange adsorption method.⁸ The use of mesoporous silica as an adsorbent has the disadvantage of poor separation between pulp and adsorbent due to the fine particle size of the adsorbent⁸ and low adsorption capacity at high pH value (pH > 9).¹⁴ Electrowinning has the disadvantages of high energy consumption with low current efficiency due to the side reactions, and cyclic utilization of the thiosulfate solution is difficult.⁸

Among purification techniques, solvent extraction¹⁵ can be the best choice for gold recovery especially if the developed extraction system has high loading capacity of extractant, high aqueous to organic ratio (A/O) in extraction step, high organic to aqueous (O/A) ratio in the stripping step, and recyclability of the extractant in order to produce highly concentrated gold solution with less generation of waste. Most of the solvent extraction studies have been done in chloride solution,^{16–22} however, only a few reports are available for the solvent extraction of the gold from thiosulfate solutions using conventional extractants.^{3,13,23}

Lately, ionic liquids have emerged as novel extractants and are playing a major role in the separation processes research including solvent extraction and membrane separation due to their high thermal/chemical stabilities, high solvent capacity, negligible vapor pressure, nonflammability, and high extraction power for metal ions.^{24–26} Ionic liquids are salts in which cation and anion are poorly coordinated and are liquid below 100 °C.²⁵ Ionic liquids are considered as potential and selective extractants for the separation of heavy metal ions from different aqueous media.²⁷ Ionic liquids have gained attention for the extraction of gold over conventional extractants from different media. Imidazolium,²⁸ pyridine,²⁹ amine,²² and phosphonium ionic liquids^{18,21} have been explored for the extraction of gold from chloride and cyanide medium.^{30,31} Phosphonium ionic liquids, with properties such as high thermal stability, high hydrophobicity, unique miscibility behavior, and fast extraction kinetics over other ionic liquids,³² have gained attention as the emerging extractants for metal extraction and have also been used for gold extraction from chloride medium.^{18,21} Fraser and Macfarlane³³ reported a list of commercially available phosphonium ionic liquids with their properties. These ionic liquids are also significantly explored for the extraction and separation of various metal ions in the past decade.³⁴

Currently, there is no reported literature on the extraction of gold from thiosulfate medium using phosphonium ionic liquids. On the basis of their application in solvent extraction, it is decided to explore phosphonium ionic liquids for the extraction of gold from thiosulfate medium. In this work, two phosphonium ionic liquids namely Cy IL 101 (tetradecyl-(trihexyl) phosphonium chloride) and Cy IL 102 (tetradecyl-(trihexyl) phosphonium bromide) diluted in toluene have been used as extractants for the extraction of gold from thiosulfate medium. The stoichiometry of the gold complex extracted and extraction mechanisms have been identified. In addition, gold stripping from loaded ionic liquid phases was investigated, and the reusability of the ionic liquid phase was confirmed up to five extraction-stripping cycles. The optimized results were extended to optimize the economic approach for gold extraction from thiosulfate medium.

EXPERIMENTAL SECTION

Materials. The reagents and chemicals used in this study were procured from Sigma-Aldrich, USA. Cy IL 101 and Cy IL 102 (see Tables S1 and S2 in the Supporting Information) were received from Cytec Canada, Inc. and were diluted in toluene to prepare a solution of desired concentration. Dilute sodium hydroxide solution was used to adjust the pH. All solutions and dilutions were prepared in deionized water. A standard gold solution (1000 mg/L) from Sigma-Aldrich, USA, with 99.999% purity was used to prepare a gold thiosulfate solution. First, the desired volume of standard gold solution was evaporated to dryness to remove the acid,³⁵ and then 0.2 mol/L sodium thiosulfate solution (Unless mentioned otherwise) with pH = 9.1 was added to prepare the Au(S₂O₃)₂³⁻ solution with a final pH = 9.0 ± 0.1.

General Methods. For partition studies, aqueous and organic phases were shaken using the desired phase ratio (A/O) at 25 ± 1 °C for 10 min to ensure equilibrium. After phase separation, raffinate was analyzed for gold concentration using atomic absorption spectrometer (AAS), model iCE 3300, Thermo Fisher Scientific. Mass balance calculations were used to determine the gold concentration in aqueous and ionic liquid phases after extraction or stripping. The percentage extraction of gold was calculated by using the following formula (eq 1):

$$\% E = \frac{D}{D + \left(\frac{V_{\text{aq}}}{V_{\text{org}}}\right)} \times 100 \quad (1)$$

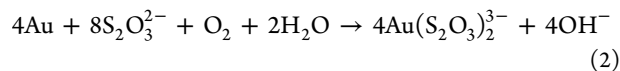
where V_{aq} and V_{org} are the volumes of aqueous and ionic liquid phases, respectively. Distribution ratio (D) was calculated using the ratio of the concentrations of metal in the organic and aqueous phase at equilibrium.

Stripping studies were also carried out by shaking the suitable stripping solution of desired concentration with the loaded ionic liquid phase (A/O = 1/1) at room temperature for 10 min (unless mentioned otherwise). The back extraction of gold, i.e., stripping, was calculated by multiplying the ratio of concentrations of the metal in the stripped phase over the loaded organic phase by a hundred. The experiments were performed in replicates, and a standard deviation of less than ±5% was found throughout the study.

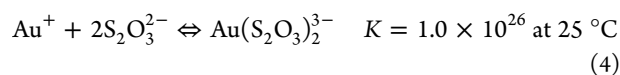
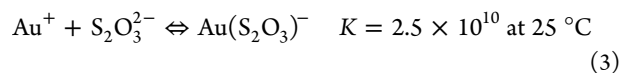
Speciation. Medusa software³⁶ with its database has been used to compute gold species in the aqueous thiosulfate solution. The extraction of Au(S₂O₃)₂³⁻ species to the ionic liquid phase was also confirmed by determining the consumption of the thiosulfate concentration in the process using iodimetric titration.³⁷

RESULTS AND DISCUSSION

Speciation of Gold in Thiosulfate Medium. The speciation of gold in the thiosulfate medium depends on the dissolution process which involves oxygen as an oxidant and thiosulfate as a ligand³⁸ (eq 2).



The peripheral sulfur atom in thiosulfate helps in making strong σ -bonds with the metal ions which are stabilized by $p\pi$ - $d\pi$ back-bonding.³⁹ Gold is mainly present in aqueous thiosulfate solutions as Au(S₂O₃)⁻ and Au(S₂O₃)₂³⁻ complex ions (eqs 3 and 4) depending on the concentration of gold and thiosulfate in solution and pH of the medium.³⁹



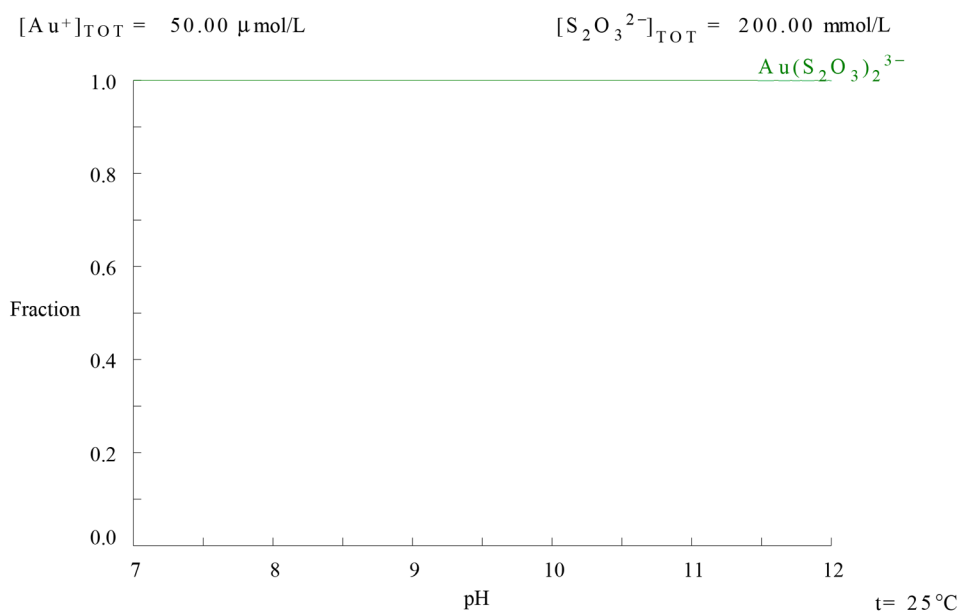


Figure 1. Speciation diagram for Au(I) species in thiosulfate medium. Conditions: $[\text{Au(I)}] = 10 \text{ mg/L}$, and $[\text{Na}_2\text{S}_2\text{O}_3] = 0.2 \text{ mol/L}$.

Figure 1 shows the fraction of gold(I) species in aqueous thiosulfate solutions for a total gold(I) concentration of 10 mg/L, and 0.2 mol/L sodium thiosulfate concentration at pH values varying from 7 to 12.

It is clear from the speciation diagram that gold(I) exists only as $\text{Au}(\text{S}_2\text{O}_3)_2^{3-}$ in the investigated range of pH and is favorable for solvent extraction using ionic liquids as an anion exchanger.

Effect of Ionic Liquid Concentration. Figure 2 represents the extraction efficiency of gold with two different

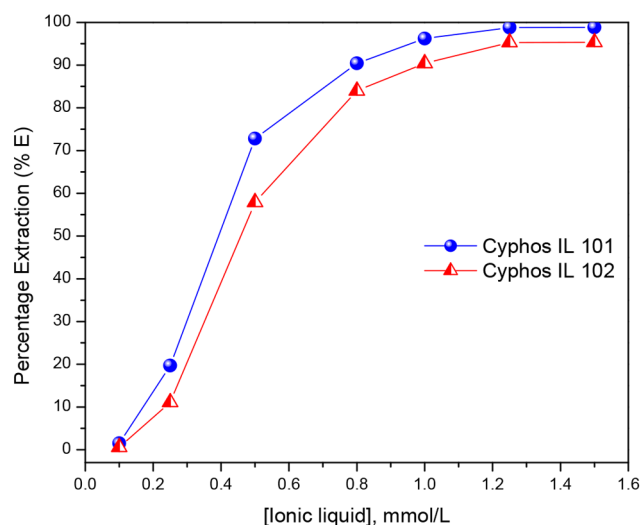


Figure 2. Influence of ionic liquid concentration on Au(I) extraction. $[\text{Ionic liquid}] = 0.1 \text{ mmol/L}$ to 1.5 mmol/L ; aqueous = 10 mg/L Au(I) ; $[\text{Na}_2\text{S}_2\text{O}_3] = 0.2 \text{ mol/L}$; $\text{A/O} = 2$; $t = 10 \text{ min}$.

ionic liquids namely Cy IL 101 and Cy IL 102. The gold extraction efficiency is positively and sharply affected by an increase in ionic liquid concentration in the organic phase. The concentration of each ionic liquid in toluene was varied from 0.1 mmol/L to 1.5 mmol/L while keeping the other

operational parameters constant, i.e., $\text{pH} = 9.0$, $\text{A/O} = 2$ and 0.2 mol/L $\text{Na}_2\text{S}_2\text{O}_3$.

Cy IL 101 showed better extraction efficiency for gold in comparison to Cy IL 102. It is due to the fact that the bromide ion in Cy IL 102 is more hydrophobic than the chloride ion present in Cy IL 101,^{40,41} hence, slowing down the process of anion exchange thereby reducing gold extraction efficiency. An extraction efficiency of 99% and 95% was achieved with 1.25 mmol/L Cy IL 101 and 1.25 mmol/L Cy IL 102, respectively. Thereafter, plateau is reached for extraction efficiency with the further increase in concentrations of Cy IL 101 and Cy IL 102. There was no significant change in pH after extraction, and no third phase or stable emulsion formation observed during the experiments. For further studies, 0.5 mmol/L extractant concentration was selected for Cy IL 101 and Cy IL 102.

Extraction Isotherm. The extraction isotherm for gold based on 0.5 mmol/L Cy IL 101 or Cy IL 102 in the organic phase and 10 mg/L Au(I) in 0.2 mol/L sodium thiosulfate aqueous phase, with an initial $\text{pH} = 9.0$ and extraction time 10 min, is presented (Figure 3) to understand the extraction efficiency of gold at different A/O ratios (1/3 to 5/1) and to evaluate saturation capacity of each ionic liquid.

Figure 3 shows that gold extraction decreases with an increasing A/O ratio for both extraction systems due to the decrease in availability of free extractant to extract gold in each case. It was observed that gold extraction decreased from 100% ($\text{A/O} = 1/3$) to 74% ($\text{A/O} = 2/1$) and 61% ($\text{A/O} = 2/1$) for Cy IL 101 and Cy IL 102, respectively. Thereafter, a further increase in the phase ratio results in a decline of extraction, and the trend of data lines in the plot is reversed. The maximum loading of organic phases (Cy IL 101 and Cy IL 102) occurred at $\text{A/O} = 2/1$, and the saturation capacity calculated was found to be 0.164 and 0.128 mol gold per mole of the Cy IL 101 and Cy IL 102, respectively. The saturation capacity data was also confirmed by equilibrating the same extractant phase with a fresh aqueous phase at $\text{A/O} = 1/1$ each time until the saturation was achieved.

Mechanism of Extraction. The mechanisms of gold extraction using Cy IL 101 and Cy IL 102 diluted in toluene

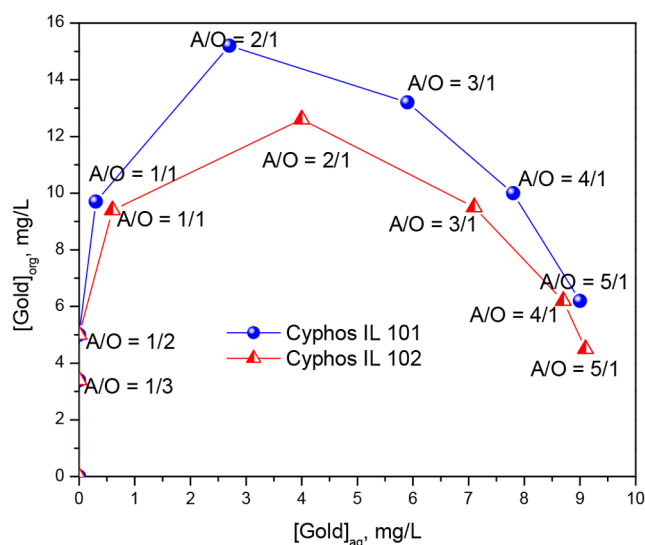


Figure 3. Effect of phase ratio (A/O) on Au(I) extraction. [Ionic liquid] = 0.5 mmol/L; aqueous = 10 mg/L Au(I); $[\text{Na}_2\text{S}_2\text{O}_3] = 0.2$ mol/L; A/O = 1/3 to 5/1; $t = 10$ min.

have been investigated. It is mentioned in the previous section that $\text{Au}(\text{S}_2\text{O}_3)_2^{3-}$ is the dominating species in the investigated range of experimental parameters. Slope analysis method⁴² was used to determine the stoichiometry in between gold extractable species and extractant molecules. On the basis of the data obtained from the effect of ionic liquid concentration, plots between $\log D$ versus $\log[\text{ionic liquid}]$ have been drawn for both ionic liquids (Figure 4).

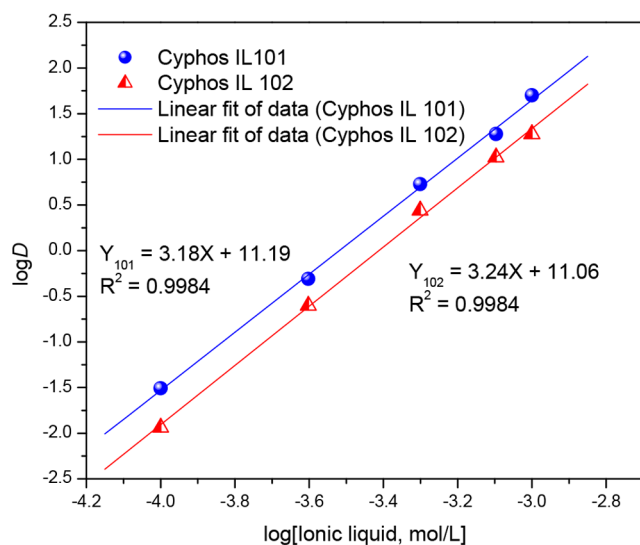
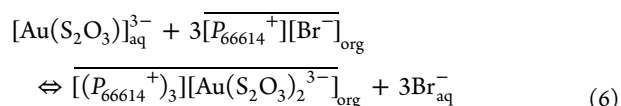
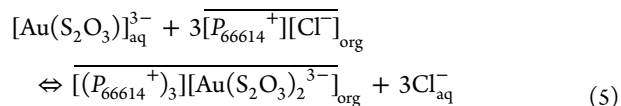


Figure 4. Slope analysis for the determination Au(I) extraction stoichiometry. [Ionic liquid] = 0.1 mmol/L to 1.5 mmol/L; $[\text{Au}(\text{I})] = 10$ mg/L; $[\text{Na}_2\text{S}_2\text{O}_3] = 0.2$ mol/L; A/O = 2; $t = 10$ min.

The straight lines with slope values equal to 3.18 and 3.24 for Cy IL 101 and Cy IL 102 extraction systems confirm the engagement of 3 molecules of Cy IL 101 or Cy IL 102 per gold ions present in the extractable complex. A stoichiometric ratio of 1:3 (metal species:ionic liquid) has been obtained for each ionic liquid diluted in toluene.

Iodimetric titration³⁷ was also performed to determine the thiosulfate concentration in the initial (before extraction) and final (after extraction) aqueous phase. A decrease in thiosulfate concentration after extraction was observed which confirms the extraction of gold as gold thiosulfate complex species. The gold extraction mechanism using Cy IL 101 and Cy IL 102 can be represented as eqs 5 and 6:



Effect of Equilibration Time. The optimum equilibration time for the distribution of Au(I) into Cy IL 101 and Cy IL 102 diluted in toluene was determined by varying shaking time from 1 to 20 min. Results reveal (Figure 5) that 10 min is enough to achieve maximum gold extraction in both ionic liquids and suggests the fast kinetics of extraction.

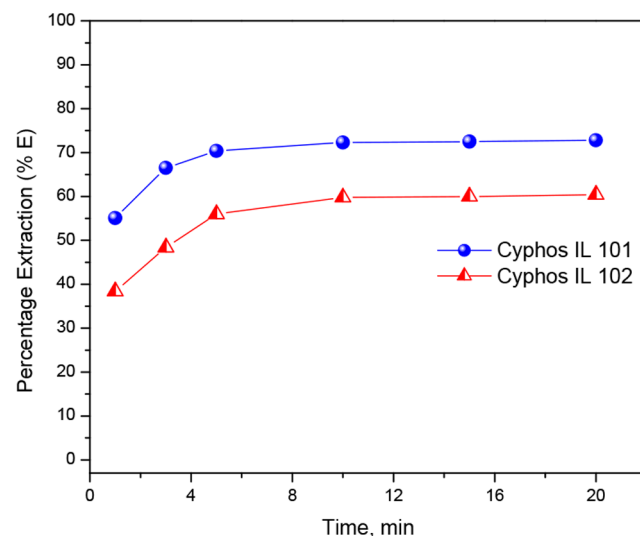


Figure 5. Effect of equilibration time on Au(I) extraction. [Ionic liquid] = 0.5 mmol/L; aqueous = 10 mg/L Au(I); $[\text{Na}_2\text{S}_2\text{O}_3] = 0.2$ mol/L; A/O = 2. Further rise in shaking time did not affect the results. In all further studies, 10 min was used as the optimized shaking time.

Stripping Studies. Stripping is the back extraction of metal ion of interest from a loaded extractant phase to a suitable aqueous phase and is necessary for the recycling of ionic liquid phase.¹⁵ An aqueous phase containing 10 mg/L gold in 0.2 mol/L sodium thiosulfate solution at pH = 9.0 was equilibrated with 1 mmol/L of each ionic liquid at O/A = 1 to generate sufficient loaded organic phases for stripping studies. Deionized water, ammonia, thiourea, and thiourea with HCl and NaCl were used as stripping agents. Each stripping test was performed using O/A = 1 at 25 °C for 10 min, and the results are represented in Table 1. The gold stripping from both ionic liquid phases with water and 1.0 mol/L ammonia solution was found negligible due to the formation of strong ionic interaction in the organic phase. The stripping of gold with thiourea or acidic thiourea (10%) showed positive results,

Table 1. Effect of Different Stripping Agents on Gold Recovery from Loaded Ionic Liquids

loaded organic phase	stripping agent	water	1.0 mol/L NH _{3(aq)}	10% thiourea	10% thiourea in 0.01 mol/L HCl	1.0 mol/L NaCl
Cyphos IL 101	% stripping	–	–	36 ± 1	65 ± 1	97 ± 2
Cyphos IL 102	% stripping	–	–	40 ± 1	69 ± 1	92 ± 2

but stripping efficiency was low probably due to high stability of the gold thiosulfate complex in the ionic liquid phase. Sodium chloride (1.0 mol/L) was found to be the promising option for the quantitative recovery (>90%) of gold from the loaded Cy IL 101/Cy IL 102 and was selected for further stripping studies.

Figure 6 shows that the stripping of gold increases sharply with rise in NaCl concentration and a stripping efficiency of

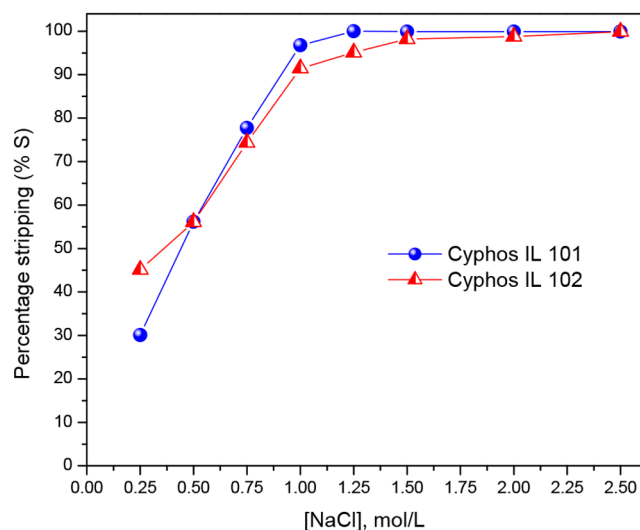


Figure 6. Effect of NaCl concentrations on the gold stripping. [loaded ionic liquid] = 1.0 mmol/L; stripping agent = 0.25–2.5 mol/L NaCl; A/O = 1.

100% and 98% was achieved using 1.5 mol/L NaCl solution for Cy IL 101 and Cy IL 102, respectively. A further increase in NaCl concentration did not influence the stripping efficiency significantly.

Recyclability of Extraction Systems. Commercial viability of an extractant system depends on its reusability.¹⁵ Recyclability of the extractant is an important aspect for its commercial use. The recyclability of Cy IL 101 and Cy IL 102 was checked up to five extraction-stripping cycles using the same ionic liquid phases each time. For this purpose, 0.5 mmol/L of each ionic liquid was contacted with 10 mg/L Au(I) in 0.2 mol/L thiosulfate medium (pH = 9.0) at A/O = 1. The loaded ionic liquid phases were stripped with 1.5 mol/L NaCl solution, and the barren ionic liquid phases were washed with deionized water for further extraction of Au(I) from a fresh aqueous phase. A change of ±4.5% and ±5.6% was observed in percentage extraction using Cy IL 101 and Cy IL 102, respectively. It is worth mentioning that recyclability of ionic liquid Cy IL 102 probably will cause the disappearance of bromide ion by its replacement with chloride ion, producing the same IL as Cy IL 101.

Scale up of Developed Process. To increase economic and environmental benefits, the developed process was scaled in such way that reduces the volume of extractants used in the extraction stage by increasing the A/O ratio and concentrated

gold solution production in the stripping stage by increasing O/A ratio generation in the stripping stage. In the Cy IL 101 extraction system, 10 mg/L Au(I) in 0.2 mol/L thiosulfate medium (pH = 9.0) was equilibrated with 3.5 mmol/L Cy IL 101 at A/O = 10 and then stripped with 2.0 mol/L NaCl at O/A = 1 in two stages. Similarly, for the Cy IL 102 extraction system, 3.5 mmol/L Cy IL 102 was used under similar experimental conditions unless mentioned otherwise. The results of both extraction systems showed promising generation of concentrated gold bearing solution (Table 2 and 3) and can be extended to the pilot scale with the addition of some experiments.

Table 2. Results of Gold Extraction from Thiosulfate Medium

stream	A/O	[Au(I)], mg/L	% extraction
feed		10.0	
loaded 3.5 mmol/L Cyphos IL 101	10/1	98.4 ± 3.2	98
loaded 3.5 mmol/L Cyphos IL 102	10/1	96.4 ± 3.8	96

Table 3. Results of Gold Stripping from Loaded Ionic Liquid Phases

stream	O/A	[Au(I)], mg/L	% stripping
loaded 3.5 mmol/L Cyphos IL 101		98.4 ± 3.2	
loaded 3.5 mmol/L Cyphos IL 102		96.4 ± 3.8	
strippant for Cyphos IL 101 (2.0 mol/L NaCl)	10/1	968 ± 13	98
strippant for Cyphos IL 102 (2.0 mol/L NaCl)	10/1	949 ± 14	98

A flowsheet has been designed for the developed process and shown in Figure 7. Applicability of the process at high A/

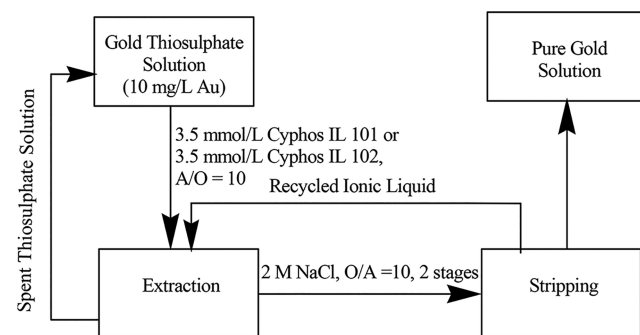


Figure 7. Flowsheet for the developed gold thiosulfate-SX process.

O ratio for extraction and high O/A ratio for stripping and reusability of ionic liquids make the process sustainable and efficient for the recovery of gold from the thiosulfate medium.

CONCLUSION

The results revealed that efficient extraction and quantitative recovery (>98%) of gold is achieved from thiosulfate solutions

with minimum production of waste in the end of the process. Ionic liquids worked as an anion exchanger for gold extraction from the thiosulfate medium, and Cy IL 101 showed better extraction capacity in comparison to the Cy IL 102 due to the difference in hydrophobicity of their anions. Gold(I) was extracted into the ionic liquids phases as $[\{P_{66614}^+\}_3\{Au(S_2O_3)_2^{3-}\}]$ and can easily be stripped using the 1.5 mol/L NaCl solution. The recyclability of the ionic liquids showed their potential for the gold extraction from the thiosulfate solution up to several cycles.

Overall, the utility of this process significantly improves the recovery of gold with economic and environmental benefits through recyclability and recirculation of the reagents used. The scale up of the process at high A/O = 10 and O/A = 10 ratios in extraction and stripping stages, respectively, also showed promising recovery of gold. The proposed procedures have industrial potential and can be extended from bench to plant scale after conducting a few additional experiments. Nevertheless, change in composition of thiosulfate leach solution with the presence of elements, e.g., Cu or Co, would need adjustment in the proposed process based on the leach solution composition and is under investigation for future research.

■ ASSOCIATED CONTENT

Supporting Information

The Supporting Information is available free of charge at <https://pubs.acs.org/doi/10.1021/acssuschemeng.1c01705>.

Chemical structure and properties of Cyphos IL 101 (Table S1) and chemical structure and properties of Cyphos IL 102 (Table S2) (PDF)

■ AUTHOR INFORMATION

Corresponding Authors

Harshit Mahandra – *Hydrometallurgy and Environment Laboratory, Robert M. Buchan Department of Mining, Queen's University, Kingston, Ontario K7L 3N6, Canada;*
ORCID: orcid.org/0000-0001-7089-2997; Email: hm90@queensu.ca

Ahmad Ghahreman – *Hydrometallurgy and Environment Laboratory, Robert M. Buchan Department of Mining, Queen's University, Kingston, Ontario K7L 3N6, Canada;*
ORCID: orcid.org/0000-0002-3568-7880; Email: ahmad.g@queensu.ca

Author

Fariborz Faraji – *Hydrometallurgy and Environment Laboratory, Robert M. Buchan Department of Mining, Queen's University, Kingston, Ontario K7L 3N6, Canada;*
ORCID: orcid.org/0000-0002-0620-2020

Complete contact information is available at: <https://pubs.acs.org/10.1021/acssuschemeng.1c01705>

Notes

The authors declare no competing financial interest.

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