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Selective extraction of gold (III) from hydrochloric acid–chlorine gas leach solutions of copper anode slime by tri-butyl phosphate (TBP)

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Abstract: The oxidative leaching causes to dissolve various impurities such as Fe, Cu, Pd, Se in copper anode slime. Organic extractant tri-butyl phosphate (TBP) was used to purify leach solution. Several parameters, such as TBP, HCl and chloride inorganic salt concentrations were chosen in order to determine efficient state for impurities separation. Standard solvent extraction tests for extraction and separation of Au, Pd, Pt, Fe, Cu and Se were conducted with equal volume of aqueous and organic phases in batch experiments. The effect of hydrochloric acid, organic phase and metals initial concentration were examined at ambient temperature. It was found that 0.25 mol/L TBP in the presence of 2.5 mol/L hydrochloric acid can cause high extraction of gold. Meanwhile, in these conditions the extraction of other impurities is negligible. After extraction, pregnant organic phase was scrubbed by distilled water and some impurities were removed. Finally, gold was stripped by sodium thiosulfate solution. The stripping solution does not have any impurities. By adding H_2SO_4 to stripping solution containing $Na_2S_2O_2$, SO_2 gas is released and Au^{3+} ions could be reduced.

Key words: gold; solvent extraction; separation; copper anode slime; TBP

1 Introduction

Recently, demands for gold and other precious metals have increased. New source identity and alteration recycling processes are the best strategies to satisfy these requests. Copper anode slime is one of the main sources for precious metals that has significant value of Au, Pt, Pb, Se, Ag, Th, etc [1].

Two main methods (e.g., pyrometallurgy and hydrometallurgy) were considered to extract precious metal from copper anode slime [2–7]. In recent years, hydrometallurgical process has been considered for precious metal recovery from copper anode slime that is solved at appropriate media along with a strong oxidizer [8–10]. Strong oxidizing agents lead to precious metals and impurities dissolution.

Solvent extraction is a suitable method for platinum group metals (PGMs) removal from low concentrated sources [11]. Our investigation [12] has shown that very low concentration of tri-butyl phosphate (TBP) could extract $HAuCl_4$ from sensitized chloride solution. Extraction percentage of Au increased along with an increase in extractant and acid concentration. But TBP was also suggested for iron [13], selenium and tellurium [14], copper and other base metals [15],

palladium and platinum [16] extraction from chloride media. Hence, it seems that gold extraction may interface with other metal species extraction.

The aim of the present work was to develop a simple, efficient and environmentally friendly process for the separation and recovery of Au from chloride leach liquors of copper anode slime by commercial phosphorus based extractant. In the previous studies, only efficient extraction for target metal has been investigated and it has determined other impurities extraction, but in this study gold and impurities extraction was studied, individually. Then, by comparing different metals extraction by TBP, efficient condition for Au extraction and purification has been introduced. Practical parameters for extraction process include extractant, acid and chloride salt concentration on gold and impurities extraction. After impurities separation studies on extraction stage, scrubbing and stripping of organic phase were surveyed for higher purification. Finally, a flow sheet for Au separation from impurities (Fe, Cu, Pd and Se) was proposed.

2 Experimental

$PtCl_6 \cdot 6H_2O$, $PdCl_6$, SeO_2 and $FeCl_3$ (Merck, Chemical Company GmbH, Germany) were used for

platinum, palladium, selenium and iron sources in aqueous solution, respectively. Selenium source was prepared by SeO_2 dissolution in dilute hydrochloric acid solution. Analytical grade of tri-*n*-butyl phosphate (FLUKA, Switzerland) as the extractant and commercial grade kerosene (Isfahan Refinery Co.) as the diluent were used for preparing stock organic solutions. All other chemicals were A.R. grade. The copper anode slime was purchased from National Iranian Copper Industries Company (NICICO).

The copper anode slime was leached by chlorination processes in the presence of hydrochloric acid and chlorine gas. The elemental composition of leach liquors is shown in Table 1. As seen in Table 1, there is gold along with other impurities, such as copper, iron, palladium, platinum in leach liquors. Hence, the homogeneous solutions of metal chlorides, e.g., gold (500 mg/L), iron (1000 mg/L), palladium (170 mg/L) and selenium (680 mg/L) with 0.05–7 mol/L HCl were prepared. Metallic salts were dissolved in distilled water and then certain amount of this solution was diluted by hydrochloric acid and distilled water.

Table 1 Concentration of metals and their variation in Sarcheshmeh anodic slime leach liquor (mg/L)

Au	Fe	Se	Cu	Ag	Pd
300–550	860–1250	800–1650	700–860	10–60	30–120

Batch experiments were conducted in a flask containing equal volume (20 mL) of aqueous, mixture of hydrochloric acid and definite initial concentration of Pd, Pt, Fe, Cu and Se, and organic phases (0.05–0.25 mol/L TBP) at ambient temperature. The mixture was mechanically agitated (400 r/min) for 30 min. The mixtures were then separated by a decanting funnel for 15 min to allow complete separation. The metal content of the aqueous phase was determined by atomic absorption spectroscopy (AAS, Varian, A240) and induction coupling plasma optical emission spectroscopy (ICP-OES, Varian, 715-ES), and then the organic phase was estimated by mass balance calculations. In some cases, the loaded organic (LO) phases were scrubbed or stripped by water and other appreciated solution, and the strip solution was analyzed for metal contents by ICP-OES/AAS. Acid content of the samples was determined by NaOH solution in the presence of phenolphthalein as the indicator. Stripping and scrubbing experimental activity was done as a simulation in experimental activity.

3 Results and discussion

3.1 Effect of TBP concentration on co-extraction of gold with impurities

The concentration of organic extractant is one of the

most important parameters on extraction of main metal and impurities. Therefore, the effect of TBP concentration on the extraction of gold from the obtained leach liquor of copper anodic slime at optimum concentration of hydrochloric acid with different concentrations (0.01–0.25 mol/L) of TBP was investigated. According to our previous work [12], to study the effect of extractant concentration on extraction of Au, the concentration of HCl in the feed solution was determined to be 3 mol/L. Experiment was performed individually for gold, iron, copper and palladium. Figure 1 shows the extraction behavior of Au and other metals as a function of TBP concentration. As seen in this figure, gold was efficiently extracted in 0.175 mol/L TBP, but at this concentration, Fe, Pd and Cu also co-extracted 21%, 23% and 11%, respectively. It seems that gold extraction from other impurities can be done in a relatively selective manner and by using TBP solvent diluted kerosene. So, separation of gold from other metals with TBP at concentration greater than 0.25 mol/L could be favorable.

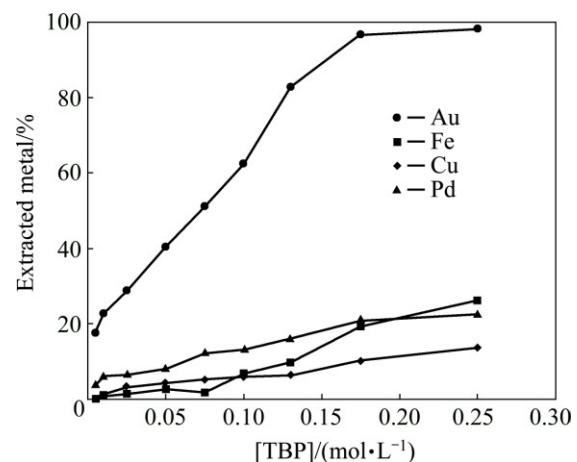


Fig. 1 Influence of TBP concentration on Au(III), Fe(II), Cu(II) and Pd(II) extraction from leach liquor (Au 500 mg/L, Fe 860 mg/L, Cu 610 mg/L, Pd 71 mg/L, HCl 3 mol/L, 25 °C)

In Table 2, the effect of TBP concentration at 3 mol/L HCl and ambient temperature on the distribution and separation factors of gold from other impurities is shown. By increasing the concentration of TBP, the distribution factor of all metals increases. However, the distribution factor of all metals, except gold, in all of TBP concentration is less than 1. In the case of gold, the distribution factor in the TBP concentrations greater than 0.075 mol/L is more than 1. Therefore, it is expected that for optimizing the extraction of gold, the TBP concentration should be greater than 0.075 mol/L. On the other hand, as can be seen in Table 2, the separation factor of gold from iron, copper and palladium changes by increasing the TBP concentration; in fact, it first decreases and then increases steadily. According to the

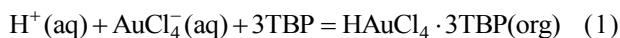
data presented in Table 2, the separation factor of gold from other metals in all the TBP concentrations is greater than 1. So, it is predicted that if the TBP concentration is greater than 0.1 mol/L, the gold extraction from other metals becomes a better separation. But it must be considered that in this case, in order to achieve higher purity of gold, the scrubbing process of extracted organic phase before stripping is recommended.

Table 2 Distribution factor (D) of some metals and separation factor (β) of gold from other metals by different concentrations of TBP at 3 mol/L HCl and ambient temperature

TBP/ (mol·L ⁻¹)	D				β		
	Au	Fe	Cu	Pd	Au/Fe	Au/Cu	Au/pd
0.005	0.215	0.001	0.003	0.038	179.167	63.235	5.628
0.010	0.294	0.011	0.011	0.066	26.241	26.009	4.453
0.025	0.406	0.014	0.035	0.070	28.194	11.667	5.767
0.050	0.678	0.028	0.047	0.089	24.669	14.496	7.657
0.075	1.052	0.019	0.055	0.141	54.797	19.129	7.467
0.100	1.660	0.073	0.063	0.151	22.865	26.266	10.993
0.130	4.797	0.109	0.069	0.192	43.889	69.523	25.037
0.175	30.746	0.240	0.114	0.262	128.108	269.229	117.531
0.250	54.556	0.355	0.159	0.294	153.591	342.686	185.437

3.2 Effect of Cl⁻ ion on extraction of gold

According to SAEEDI et al's [17] discussion on the dissolution of copper anode slime, it is shown that there is a stable increase in the concentration of chloride ion composition AuCl_4^- . SADEGHI et al [12] with respect to the mechanism of gold extraction proposed the following reaction:



On the other hand, SADEGHI and ALAMDARI [18] claimed that by changing the concentration of chlorine ion, the oxidative conditions of AuCl_4^- ion stability changes:



It seems that, decreasing Au concentration increases AuCl_4^- stability in thermodynamic viewpoint. Thus with regarding the dynamic decrease of Au concentration in aqueous solution, the AuCl_4^- was known as dominate species in solvent extraction process.

Thus, the effect of chlorine ion on the extraction of gold by 0.25 mol/L TBP from aqueous media, containing 3 mol/L hydrochloric acid at ambient temperature, was studied. The chlorine ion concentration of NaCl was changed. The results are shown in Fig. 2. As seen in the figure, increasing the concentration of NaCl has no

significant effect on gold extraction, and even with salt concentration more than 1 mol/L, it slightly decreased. Therefore, an increase in chlorine ion alone does not have any significant effect on gold extraction while the concentration of hydrochloric acid is one of the important factors in gold extraction. As noted, the studies conducted by SADEGHI and ALAMDARI investigating the effect of increasing of HCl concentration on gold extraction were located. Increasing of chlorine ion concentration changes the stability of Se, Fe and Cu ionic species. Accordingly, the ionic species stability could affect the extractability of metals. So, it seems that, identifying extraction behavior of the presented metals in TBP is necessary.

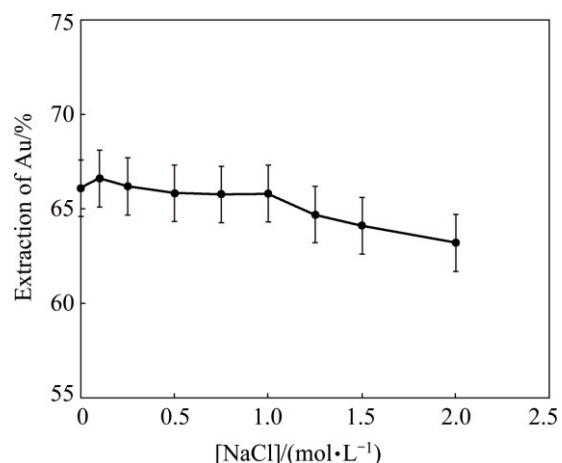


Fig. 2 Effect of chlorine ions on extraction of gold in 3 mol/L HCl, 0.25 mol/L TBP and 25 °C

3.3 Effect of HCl concentration on co-extraction of metals

3.3.1 Fe³⁺ extraction

The effect of the concentration of hydrochloric acid on extraction of impurities can significantly contribute to the improvement of the optimum acid concentration in the solvent extraction of gold. To determine the effect of acid concentration on the extraction of iron from aqueous phase, experiments with varying concentrations of 0.01 to 6 mol/L hydrochloric acid solution containing Fe³⁺ ion synthetic solution in different concentrations of TBP were done at room temperature. Figure 3 shows the Fe³⁺ ions extraction versus initial hydrochloric acid concentration in aqueous phase. As seen in the figure, the effect of an increase in the concentration of hydrochloric acid to about 2.5 mol/L, in all of TBP concentrations, on the extraction of iron is negligible. Then, a rise in the hydrochloric acid concentration up to about 3 mol/L at concentration of TBP less than 0.25 mol/L has no significant impact on the extraction of iron. An increase in the concentration of hydrochloric acid more than 3 mol/L can cause the extraction of iron to be significant. It seems that, the presence of chlorine or hydrogen ions,

or both of them, has a key role in iron extraction. According to the recent results this may have a significant impact on the concentration of Cl^- ions of iron extracted. For the determination of extraction mechanism, the effect of Cl^- on extraction of iron was studied by adding NaCl that was varied from 0 to 3 mol/L. Therefore, based on the results presented in Fig. 4, an increase in the NaCl concentration improves the efficiency of Fe extraction. While the extraction percentage increased from 35% to 54%, H^+ concentration keeps constant. In this situation, the iron cation is stable in low concentrations of chlorine ions, but an increase in chlorine ions leads to FeCl_3 formation and HFeCl_4 species could be extracted by TBP.

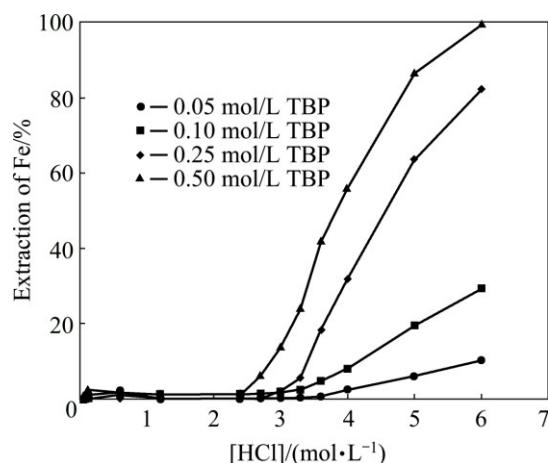


Fig. 3 Fe extraction vs HCl concentration with different TBP concentrations at ambient temperature ($[\text{Fe}^{3+}] = 1000 \text{ mg/L}$)

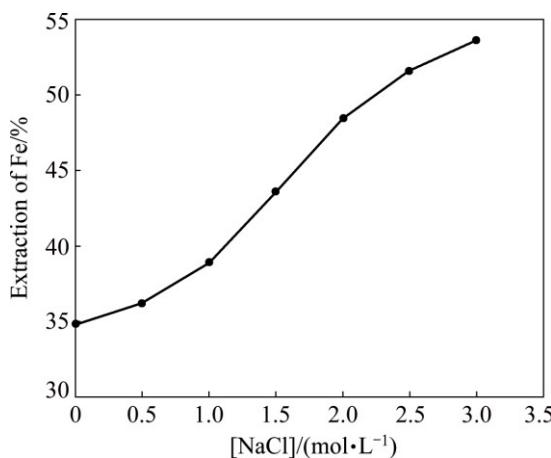


Fig. 4 Effect of NaCl concentration on Fe extraction under 4 mol/L HCl, 0.25 mol/L TBP and 25 °C

3.3.2 Palladium extraction

The same method was used for palladium extraction. The palladium and hydrochloric acid concentration in aqueous solution were selected at 190 mg/L and 1–7 mol/L, respectively. The extraction of palladium by TBP versus HCl acid concentration is depicted in Fig. 5. Unlike iron, palladium extraction decreased with an

increase in hydrochloric acid concentration. Pd extraction percentage dropped from 63% to 26% with an increase in hydrochloric acid concentration from 0.1 to 7 mol/L.

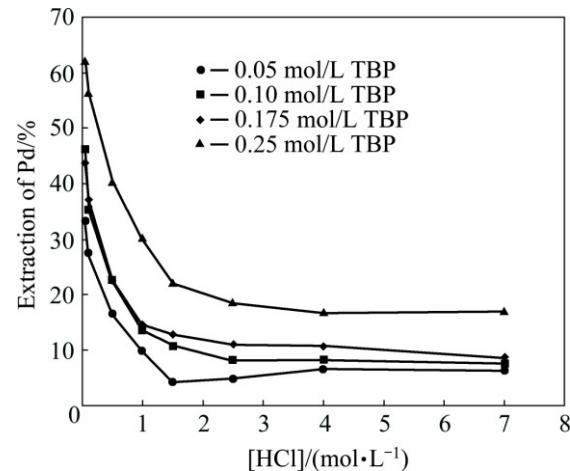


Fig. 5 Effect of HCl concentration on extraction of palladium with different TBP concentration in kerosene at ambient temperature

Palladium and Cl^- ion participate in chloride complexes formation. From thermodynamic approach, PdCl_3^- and PdCl_2^- are stable species in low ionic strength [19], but an increase in Cl^- concentration leads to an increase in ionic strength and PdCl_3^- is formed. Our investigation shows that metal species extraction percentage increased with a decrease in negative charge of ionic species.

3.3.3 Selenium extraction

To study the effect of Cl^- on selenium extraction, selenium dioxide (SeO_2) was dissolved in dilute HCl. By adding distilled water and hydrochloric acid, Se concentration reached 820 mg/L. Figures 6 shows the selenium extraction by TBP with adding different

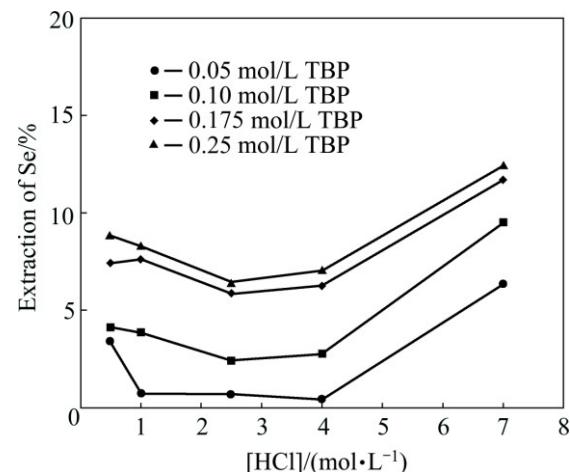


Fig. 6 Se extraction vs HCl concentration at various TBP concentrations and 820 mg/L Se at ambient temperature

concentration of HCl. Under different HCl concentrations, Se extraction percentage is under 15%. Hence, TBP can be an appropriate extractant for separating selenium from other precious metals.

3.3.4 Copper extraction

Cu extraction by TBP extractant was investigated with constant concentration of 750 mg/L Cu under different concentrations of hydrochloride acid with 0.1 and 0.25 mol/L TBP as extractant. As seen in Fig. 7, the HCl concentration is the main parameter affecting Cu extraction. The experimental data showed negligible extraction under low (1–2 mol/L) concentration of HCl but an increase in extraction from 4% to 24.4% with 0.1 mol/L TBP and 6.5% to 61.2% with 0.25 mol/L TBP with an increase in HCl concentration. Cu can form chloride complexes, such as CuCl^- and CuCl with chlorine ions. At low concentration of Cl^- , cation species was stable thermodynamically. But when Cl^- concentration increases, chloride complexes and anionic species form. Hence, Cu extraction by TBP done by chloride media could be interesting.

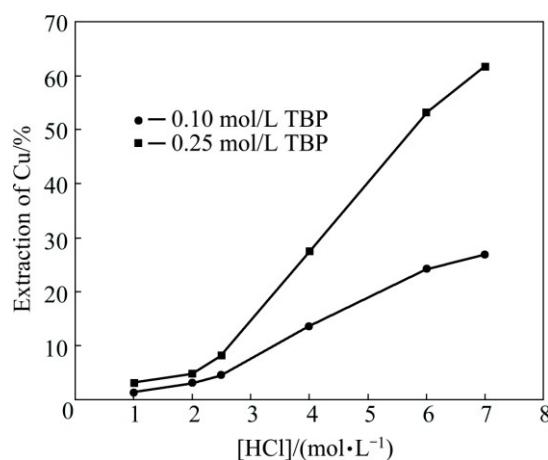


Fig. 7 Cu extraction vs HCl concentration at initial concentration of 740 mg/L Cu and ambient temperature

3.4 Extraction of metals from real leach solution

According to the test results, it can be concluded that TBP as an organic solvent is suitable for the extraction of gold at concentration of 0.1–0.25 mol/L from aqueous phase with hydrochloric acid concentration of 2.5–3 mol/L. As a result, the simultaneous extraction of Au, Fe, Pd, Se and Cu from the obtained real leaching solution could be helpful. The used aqueous solution was prepared from leach solution of Sarcheshmeh copper refining anodic sludge in the presence of 3 mol/L hydrochloric acid and chlorine gas [17]. As seen in Table 3, after one stage of extraction, the extraction of gold is more than 95% and the extraction of palladium is less than 30%. While the extraction of other metals except silver and lead is less than 10%. On the basis of thermodynamic data, the solubility of Ag and Pb in

aqueous media in the presence of chlorine ions is very low. As seen in Table 3, the presence of chlorine gas as the oxidizing agent has a little effect on the dissolution of Ag and Pb during leaching process. Through mixing and solvent extraction process, turbulent conditions of aqueous phase could cause a decrease in the dissolution of chlorine gas, dissolved in the solution, and this reduces the solubility of Ag and Pb (as well as Sn and Hg if they are present in the solution). Thus, under this condition, silver, lead, tin and mercury could be precipitated as AgCl , PbCl_2 , SnCl_2 , HgCl_2 at the bottom of the laboratory container. After completion of the solvent extraction test, some sediment was seen at the bottom of the experiment container. So, insoluble compounds are precipitated from aqueous phase and it should be considered in mixer-settler design.

Table 3 Elemental composition before and after solvent extraction (SX), distributing ratio and separation factor for gold and other impurities in chloride leach liquor and 3 mol/L HCl concentration

Element	Composition/ ($\text{mg} \cdot \text{L}^{-1}$)		Extraction/ % Before SX	Extraction/ % After SX	Distribution factor (D)	Separation factor (β)
	Before SX	After SX				
Au	455	22	95.16	19.68	—	—
Fe	1100	996	9.45	0.10	188.49	—
Se	870	850	2.30	0.02	836.48	—
Cu	742	702	5.39	0.06	345.42	—
Pd	84	59	29.76	0.42	46.45	—
Ag	25	N d.*	—	—	—	—
Pb	75	N d.*	—	—	—	—

*Not detected

As seen in Table 3 the distribution factors of all metals other than gold and palladium is less than 0.1. This amount represents the separation of gold in the presence of other impurities.

3.5 Organic solution scrubbing

As mentioned above, during the gold extraction stage by TBP in hydrochloric acid solution, minor constituents, such as Fe, Cu, were also co-extracted. Thus, in order to remove these minor impurities effectively, a scrubbing process was followed. Distilled water was used as the scrubbing agent. At the first stage, the extraction process was done with 0.25 mol/L TBP from aqueous media containing 3 mol/L HCl, and the initial and final metal concentrations in aqueous phase are shown in Table 4. At the second stage, the loaded organic phase was agitated with distilled water in $V_o:V_a=1:1$ and the concentration of stripped metals was analyzed by AAS method. As seen in Table 4, iron and

copper were readily scrubbed from the organic phase. Here the Se scrubbing efficiency was medium (62%). Hence, from the above observations, it was concluded that the scrubbing of Fe and Cu from the loaded organic phase was comparatively effective with distilled water, while Au remained in organic phase.

Table 4 Scrubbing of extracted organic phase by distilled water at room temperature ($V_o:V_a=1:1$)

Metal ion	Metal concentration/(mg·L ⁻¹)		Scrubbing percentage/%
	Organic phase (before scrubbing)	Scrubbed solution	
Au	433	4.2	428.8 0.97
Fe	104	101	3 97.12
Cu	20	18.1	1.9 90.50
Se	40	26.2	13.8 65.50
Pd	25	0.8	24.2 3.20

3.6 Stripping of Au

After scrubbing stage, the stripping process was followed to recover gold as a pure solution. There are many compounds used as stripping media for stripping of gold. Compounds such as ammonia, sulphuric acid, thiourea and sodium thiosulfate were noted. To determine the optimal conditions for stripping, the scrubbed organic solvent was washed at room temperature at $V_a:V_o=1:1$ with an aqueous solution containing compounds like ammonia, sulphuric acid, thiourea, sodium thiosulphate with different concentrations, as well as the mixture of sulphuric acid and sodium thiosulphate, in which the concentration of sodium thiosulphate remained constant at 0.1 mol/L and the concentration of sulphuric acid changed.

Table 5 shows the result of gold stripping from the loaded organic phase with various stripping agents. It is shown that sodium thiosulphate and thiourea could be

Table 5 Stripping percentages of Au with various stripping agents (Loaded organic phase containing 800 mg/L Au, $V_o:V_a=1:1$, 25 °C)

Agent	Au stripping percentage/%						
	0.01 mol/L	0.05 mol/L	0.1 mol/L	0.2 mol/L	0.25 mol/L	0.5 mol/L	1 mol/L
Ammonia	19.4	31.65	14.25	—	—	—	—
H ₂ SO ₄	1	0.9	0.81	0.69	0.5	0.5	0.15
Thiourea	86.3	89.34	91.2	91.64	92.4	—	—
Sodium thiosulfate	—	90.9	92.12	94.82	96.67	98.66	—
0.1 mol/L thiourea+ H ₂ SO ₄	—	92.7	94.2	93.6	95.38	95.7	96.2

selected as stripping agent. The stripping test was performed at ambient temperature in aqueous media containing sodium thiosulphate and thiourea. The results of chemical analysis of aqueous solution after the stripping are shown in Table 6. It can be seen in Table 6 that stripping process is well done and the amount of impurities in the solutions is negligible. On the other hand, the stripping process in aqueous media containing sodium thiosulphate is better than that in thiourea media.

Table 6 Concentration of metals after stripping with sodium thiosulfate and thiourea

Stripping solution	Concentration/(mg·L ⁻¹)				
	Fe	Se	Cu	Pd	Ag
Sodium thiosulfate	N. d.*	<5	N. d.*	N. d.*	N. d.*
Thiourea	<10	<5	<5	<20	N. d.*

* Not detected

3.7 Flow sheet of process

Based on the above mentioned results, solvent extraction by TBP is a suitable method for the recovery of gold from the leaching solutions of copper refining anodic slime. Accordingly, the flowchart of Fig. 8 is recommended for gold recovery from the leach liquor. As seen in this figure, after multi-stage extraction, TBP extracts gold and some other impurities from aqueous media containing 3 mol/L HCl at ambient temperature. After extraction, impurities in organic phase were scrubbed and removed by distilled water. With a sodium thiosulfate solution, the gold is stripped from the organic solvent. The gold present in the aqueous phase is reduced and deposited by the liberation of SO₂ gas and sulphur. The SO₂ gas is one of the products of the reaction between sodium thiosulfate and sulphuric acid (Eq. (4)). The released SO₂ gas could reduce Au³⁺ ionic species.

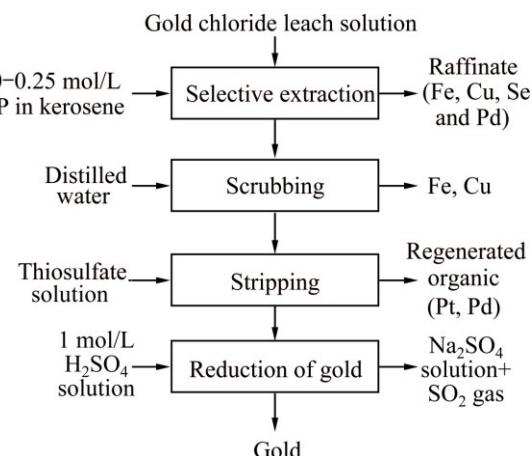


Fig. 8 Gold separation flow sheet

4 Conclusions

1) A complete hydrometallurgical process for the recovery of Au from chloride leach liquors of copper anode slime was developed by solvent extraction route using phosphate extractant TBP.

2) Practical parameters, e.g., HCl and TBP concentrations, were selected to produce a pure product with maximum efficiency. Fe and Cu extraction increases with the increase of HCl concentration, whereas Pd extraction decreases; and the extraction of selenium does not show significant changes.

3) Chloride ions in aqueous solution decrease the separation efficiency of extraction process. Thus, 0.25 mol/L TBP and 3 mol/L HCl were selected and recommended as appropriate conditions for gold extraction and impurities separation.

4) From the integrated separation experiment by the simulated solution at 25 °C, $V_0:V_a=1:1$, and one stage contact at extraction step, the extraction percentage of Au was 95.16% and 104 mg/L Fe, 20 mg/L Cu, 40 mg/L Se and 25 mg/L Pd were co-extracted to organic phase as major impurities.

5) Loaded organic phase was scrubbed by distilled water. Fe, Cu and Se were easily stripped, while Au couldn't be stripped and Fe, Cu and Se scrubbing efficiencies were 97.12%, 90.5% and 65.5%, respectively.

6) Sodium thiosulphate and thiourea+H₂SO₄ solutions were appropriate stripping agents. More than 96% of leached gold was recovered while impurities of lower than 10 mg/L for sodium thiosulphate as stripper solution were eliminated. In order to increase gold recovery, scrubbing stage can be eliminated. Gold recovery increased whereas iron and copper concentration increased in strip solution. Adding sulfuric acid into strip solution (sodium thiosulphate) and using SO₂ gas as the reducing agent gold particle was deposited.

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TBP 萃取剂从铜阳极泥盐酸-氯气浸出液中选择性萃取金

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摘要: 铜阳极泥的盐酸-氯气氧化性浸出会导致大量杂质元素如 Fe、Cu、Pd、Se 等的溶解。以 TBP 作为萃取剂, 采用标准的溶剂萃取实验方法, 等体积有机相和水相来萃取分离 Au、Pd、Pt、Fe、Cu 和 Se 等元素, 研究 TPB 浓度、HCl 和氯离子浓度对杂质分离效果的影响。结果表明, 在有机相中 TBP 浓度为 0.25 mol/L、水相中 HCl 浓度为 2.5 mol/L 的条件下, 可以得到高的金萃取率。同时, 其他杂质元素的萃取可以忽略不计。对负载有机相采用蒸馏水进行洗涤, 可以去除部分杂质元素。采用硫代硫酸钠溶液进行反萃, 反萃液中不含任何杂质元素。在反萃液中加入硫酸, 反应产生的 SO₂ 气体可还原金离子。

关键词: 金; 溶剂萃取; 分离; 铜阳极泥; TBP

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