



TECHNICAL ARTICLE

Gold Leaching from Copper Anode Slime with 1-Butyl-3-Methyl Imidazolium Chloride

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The aim of this study was to realize gold leaching from copper anode slime (CAS) with ionic liquid treatment. A novel medium, 1-butyl-3-methyl-imidazolium chloride (BmimCl), was used as the leaching reagent. The effects of ionic liquid concentration, temperature, time, and solid/liquid ratio (S/L) on the leaching efficiency were investigated by Taguchi optimization and ANOVA methods. The results showed that 61.20% of gold leaching efficiency was achieved from CAS, under the optimum leaching conditions which were determined as %80 ionic liquid concentration, 50°C temperature, 1 h time and 1/20 g/mL solid/liquid ratio. The statistical results of the experiments revealed that the effects of each parameter on the gold leaching with BmimCl were in the following order: temperature > time > solid/liquid ratio > ionic liquid concentration.

INTRODUCTION

Global demand for precious metals, especially for Au, is growing relatively faster than the requirement for other metals, which is mainly attributed to technological developments. Although the use of precious metals is generally lower than other metal sources, important problems may occur in the supply of these metals.¹ For example, gold-containing mineral resources are generally of low grade, resulting in high mining and usage costs.² Thus, secondary sources such as end-of-life printed circuit boards, spent catalysts, and other metallurgical wastes are being taken into account as very important reservoirs for precious metal production.^{3,4} Copper anode slimes (CAS) are economically attractive, and are considered as one of the important metallurgical wastes for the extraction of precious metals, such as Au, Ag, and platinum group metals. The CAS are produced as a by-product from the electro-refining process of crude copper, and also

contain many valuable metals, such as Sn, Pb, Cu, Zn, Ni, Bi, and As, which are in the form of insoluble phases.^{5,6} Copper anode slimes are regarded as refractory gold sources due to their ultra-fine gold particles (smaller than 74 μm) and the presence of gold as an insoluble compound in cyanide.⁷ As stated in previous studies,^{8,9} Au and Ag may exist in CAS as very finely dispersed in the form of selenide and telluride, because of the special affinity of elements of the IB group to the VIA group in the periodic table. It is known that the mineralogical structure of CAS is very complex, as they contain different elements. Therefore, the metal recovery method from CAS should be chosen according to the physical and chemical properties of the precious metals, and the existing forms of the various elements.^{10,11}

Until now, hydrometallurgical methods for gold recovery from CAS have been used frequently due to their easy applicability and high yields. In this method, cyanide,^{12,13} aqua regia,¹⁴ and aqueous solution of conventional acids¹⁵ were used several times for gold leaching from CAS. However, the use of these chemicals for gold leaching has begun to be limited due to their negative effects on the

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environment and human health. Therefore, ionic liquids (ILs) are being considered as novel solvents for hydrometallurgical processes because of their chemical and physical properties, such as negligible vapour pressure, being reusable, and thermal stability.^{16,17} Imidazolium-based ILs with HSO₄ anions have begun to be used for precious metal extraction from gold ore and printed circuit boards (PCB).¹⁸ Whitehead et al.¹⁹ investigated the use of some ILs based on 1-butyl-3-methyl imidazolium cations combined with different anions (CH₃SO₃, BF₄, Cl, N(CN)₂, and HSO₄) to extract gold from natural gold ore. The results of this work showed that the highest gold extraction was achieved by using 1-butyl-3-methyl-imidazolium hydrogen sulfate (BmimHSO₄), while gold leaching with 1-butyl-3-methyl-imidazolium chloride (BmimCl) and 1-butyl-3-methylimidazolium dicyanamide was very limited under the same leaching conditions (50°C, 48 h, and 1/4 g/mL).¹⁹ In addition, Whitehead et al.²⁰ reported that gold leaching from sulfidic ore with BmimCl can reach a recovery rate of 85% only by adding HSO₅⁻ as oxidant and NaI as complexing agent. Otherwise, it remains at about 30% Au recovery without any added complexing agent.²⁰ In a recent study, Barrueto et al.²¹ used BmimCl and 1-butyl-3-methyl imidazolium bromide (BmimBr) for gold leaching from PCBs. According to the results, the highest gold leaching efficiencies, under the leaching conditions of 60% v/v IL concentration, 60°C leaching temperature, 24 h leaching time, and 1/15 g/mL solid/liquid ratio, were obtained as 19.1% and 40.9% for BmimCl and BmimBr, respectively.²¹ On the other hand, initial studies on CAS^{22,23} revealed high gold extractions of 97.5% and 89.1% by using BmimHSO₄ and 1-ethyl-3-methyl-imidazolium hydrogen sulfate, respectively. Up to now, only the effects of ILs with HSO₄ anions on gold recovery from CAS were examined. However, there is no any information about imidazolium-based ILs with a different anion. Therefore, in this study, the gold leaching from CAS was investigated in a shorter leaching period with BmimCl as a novel leaching agent. Also, the leaching conditions for gold were optimized by the Taguchi method. In addition, chemical and mineralogical analyses were performed.

MATERIALS AND METHODS

Materials

CAS were used as the gold source and were provided from a copper production plant in Turkey. The CAS were ground in an axial ball mill to obtain a small particle size prior to the leaching experiments and characterization studies. BmimCl IL was used as a leaching agent, because it has a short alkyl chain which results in a significant increase in viscosity and water miscibility.²⁴ Commercial BmimCl was supplied by ABCR.

Characterization of CAS

Characterization of CAS was carried out with several methods just before the leaching experiments. In order to determine the particle size of the CAS, a Malvern Particle Sizer (Zetasizer Nano ZS) was used. Chemical analysis of CAS was realized by a fire-assaying method for Au and Ag, and by inductively coupled plasma– mass spectrometry (ICP-MS; Agilent 702) for other metals. Mineralogical structures of CAS were determined with an x-ray diffractometer (XRD; Bruker D8 Advance with Da Vinci) with Cu K α radiation at 30 kV, at a scanning rate of 0.4° min⁻¹ and field-emission scanning electron microscopy (FE-SEM; Hitachi SU5000) equipped with an energy dispersed X-ray analysis (EDX) device. In the SEM/EDX analysis, any coating was not applied. Leach residue was also investigated to reveal possible changes in the mineralogical structure of CAS after leaching of the BmimCl IL.

Leaching Experiments and Optimization Method

Leaching experiments were performed in closed flasks on a temperature-controlled heater with a magnetic stirrer. The experimental leaching parameters were selected as IL concentration, temperature, time, and solid/liquid ratio based on our previous studies.^{22,23} IL concentrations (v/v) of BmimCl were prepared with diluted water in the range of 20% to 80% with the increment of 20%. The leaching tests were carried out at a solid/liquid ratio of 1/10–1/25 g/mL, using the constant volume of leaching solution (50 ml) in the temperature range of 25–95°C. The time was selected in the range of 1–8 h. Stirring speed was kept constant as 600 rpm. After the leaching experiments, the leach residue and pregnant leach solution were separated from each other with a vacuum filtration system. Then, the leach solution was analyzed by ICP-OES. The leaching efficiency of gold was calculated by:

$$\text{Leaching efficiency (\%)} = C \times V / m \times w \quad (1)$$

where C is the average of three replicates of diluted leach solution (in mg/L), V is the volume of the leach solution (in L), m is the initial amount of anode slime (in mg), and w is the metal content of anode slime (in mg/g).

The Taguchi method, which is a simple and effective optimization method for hydrometallurgical processes,^{25,26} was used in order to determine the optimum parameters for gold recovery from CAS. Leaching parameters were investigated by using the orthogonal array (OA) experimental design method with L₁₆ (4⁴), as given in Table I with the experimental results. To maximize the gold leaching from the CAS, the performance statistics of OA were evaluated by:

$$(S/N)_L = -10 \log \left(\frac{1}{n} \sum_{i=1}^n \frac{1}{x_i^2} \right), \quad \text{larger is the better} \quad (2)$$

where $(S/N)_L$ is the performance statistic corresponding to signal noise ratios of “larger is better,” n is the number of repetitions for an experimental combination, and x_i is the performance value of the i^{th} experiment.

Collected data for gold leaching were analyzed with Minitab 17 statistical software to draw performance statistic graphs for the investigated parameters. The Taguchi method suggests performing the validation experiment under the determined optimum leaching conditions.^{27,28} For this purpose, the predicted $[S/N]_{\text{Predicted}}$ can be calculated theoretically as:

$$[S/N]_{\text{Predicted}} = [S/N]_{\text{Mean}} + \sum_{i=1}^n ([S/N]_i - [S/N]_{\text{mean}}) \quad (3)$$

where $[S/N]_{\text{mean}}$ is the arithmetical mean of the whole experimental $[S/N]_L$ ratios, $[S/N]_i$ is the arithmetical mean of the $[S/N]_L$ ratios at the optimal level for each studied parameter, and n is the number of the factor that affects the gold recovery from the CAS. Finally, analysis of variance (ANOVA) was used to determine the effect of each parameter on the optimization criteria for gold leaching.

RESULTS AND DISCUSSION

Characterization of CAS

To develop a suitable gold leaching, it was essential to determine the composition of the CAS. As demonstrated, chemical analysis of the CAS, as revealed in our previous studies,^{22,29} was mainly composed of 23.1% Cu, 20.5% Sn, and 15.6% Pb. Other elements, such as Ba, S, Ni, Sb, and Zn, were also found in the CAS as 5.9%, 4.1%, 0.8%, 0.2%, and 0.1%, respectively. According to the chemical analysis of the CAS, as an important source for precious metal extraction it contains remarkable amounts of Ag (0.22%) and Au (21.9 g/t). In addition, it was detected that the CAS includes 413 ppm of Se and 83 ppm of Te, which are valuable metals for the energy industry. The mineralogical characterization of anode slime determined by XRD and SEM/EDX analysis is shown in Fig. 1a–d.

According to the ICDD database, the major components of the CAS were PbSO_4 and SnO_2 . Also, the CAS included Cu_2O as a copper-bearing compound. Other structures included in the CAS were detected as BaSO_4 , NiO , and SbAsO_4 . As demonstrated in Fig. 1b, the CAS has small particles in different shapes. This result is very consistent with the particle size analysis due to more than half of the particles of the CAS being smaller than $23.3 \mu\text{m}$. The EDX result of the CAS (Fig. 1c) stands with its chemical analysis determined by ICP-MS.

The elemental color mapping method obtained by SEM/EDX analysis was used to reveal the possible phases in the sample. In elemental color mapping, copper is represented with red, lead by purple, tin by orange, oxygen by blue, sulfur by yellow, barium by white, and nickel by green. Thus, the distribution of the main elements (Cu, Pb, Sn, O, S, Ba, and Ni)

Table I. L_{16} (4^4) orthogonal array of Taguchi and gold leaching efficiency from anode slime

Exp. no.	Leaching parameters and levels				Gold leaching (%)	$(S/N)_L$ values
	Ionic liquid concentration (%)	Temperature (°C)	Time (h)	Solid/liquid ratio (g/mL)		
1	20	25	1	1/10	40.6 ± 1.3	32.18
2	20	50	2	1/15	37.9 ± 1.1	31.57
3	20	75	4	1/20	43.9 ± 1.2	32.85
4	20	95	8	1/25	41.5 ± 1.2	32.35
5	40	25	2	1/20	44.9 ± 1.2	33.04
6	40	50	1	1/25	60.0 ± 0.1	35.57
7	40	75	4	1/10	14.5 ± 1.2	23.20
8	40	95	8	1/15	24.8 ± 1.5	27.88
9	60	25	4	1/25	25.2 ± 1.4	28.02
10	60	50	8	1/20	44.3 ± 1.2	32.93
11	60	75	1	1/15	39.1 ± 1.3	31.85
12	60	95	2	1/10	37.1 ± 1.2	31.39
13	80	25	8	1/15	55.6 ± 1.1	34.90
14	80	50	4	1/10	45.6 ± 1.3	33.18
15	80	75	2	1/25	31.6 ± 1.3	29.98
16	80	95	1	1/20	44.3 ± 1.1	32.94

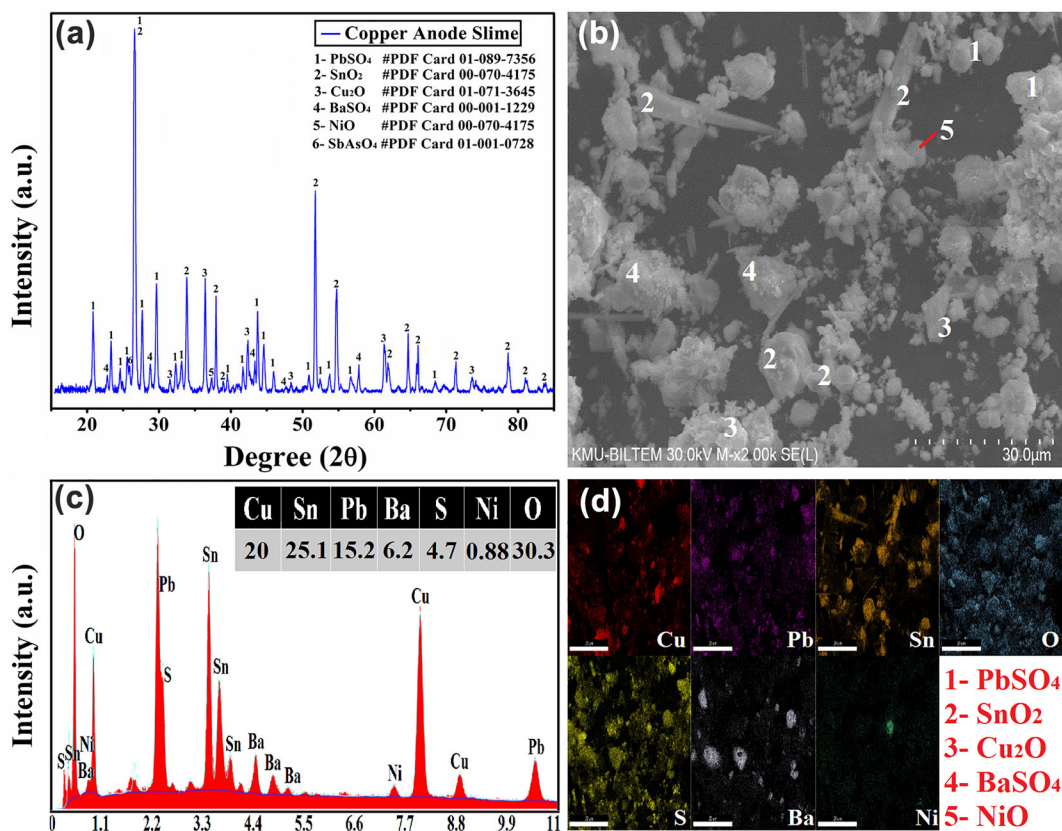


Fig. 1. (a) XRD pattern, (b) SEM image, (c) EDX pattern, and (d) elemental mapping of copper anode slime.

in CAS is illustrated in Fig. 1d. Although some of these (Cu, Pb, Sn, O, and S) are homogeneously distributed in representative SEM images, the others (Ba and Ni) have been accumulated in certain regions. When Fig 1b and d are examined together, it is seen that the labeled structures belong to different components: (1) the particles labeled 1 indicate the PbSO₄ phase, (2) the needle-type particles, labeled 2, belong to SnO₂, and (3) the Cu₂O, BaSO₄, and NiO phases contained in the CAS are marked 3, 4, and 5, respectively.

Leaching Results of CAS

Leaching tests under the conditions in Table I were performed randomly in an aqueous solution of BmimCl. The amount of gold in the solution obtained after the leaching experiments of CAS with BmimCl was determined by ICP-OES. The Taguchi L₁₆(4⁴) experimental design with the experimental results and signal/noise ratios (S/N)_L are shown in Table I.

According to the results, various gold leaching efficiencies were obtained between 14.5% to 60.0%. The (S/N)_L ratios calculated by Eq. 2 were used to draw performance statistics graphs for each parameters. The graphs for gold leaching are illustrated Fig. 2.

The highest gold leaching efficiency is substantial for the further experiments and the quality of the

gold. The optimum levels for the investigated parameters were determined according to the highest S/N ratio in the levels of those parameters. Optimum leaching conditions determined for gold leaching from the CAS and the results of the validation experiments are summarized in Table II.

By using performance statistics graphs, the optimum leaching conditions for gold leaching from CAS were determined as: IL concentration: 80%, temperature: 50°C, time: 1 h, and solid/liquid ratio: 1/20 g/mL. When the experimental conditions given in Table I are examined, it can be seen that the optimum leaching conditions are not found. Hence, the gold leaching efficiency belonging to the optimum leaching conditions must be estimated. After selecting the optimum level for each parameter, the predicted gold leaching efficiency was calculated by Eq. 3 as 63.3%. On the other hand, a validation experiment was carried out at the selected optimum conditions to verify the predicted result. As seen in Table II, the gold leaching obtained from the validation experiment was 61.2%. Considering the calculated margin error (3.3%), it can be concluded that good agreement exists between the predicted and confirmed leaching efficiencies of gold.

The performance statistics graphs used to determine the optimum parameters in the Taguchi method do not show the process effect of the investigated parameters on gold leaching from the

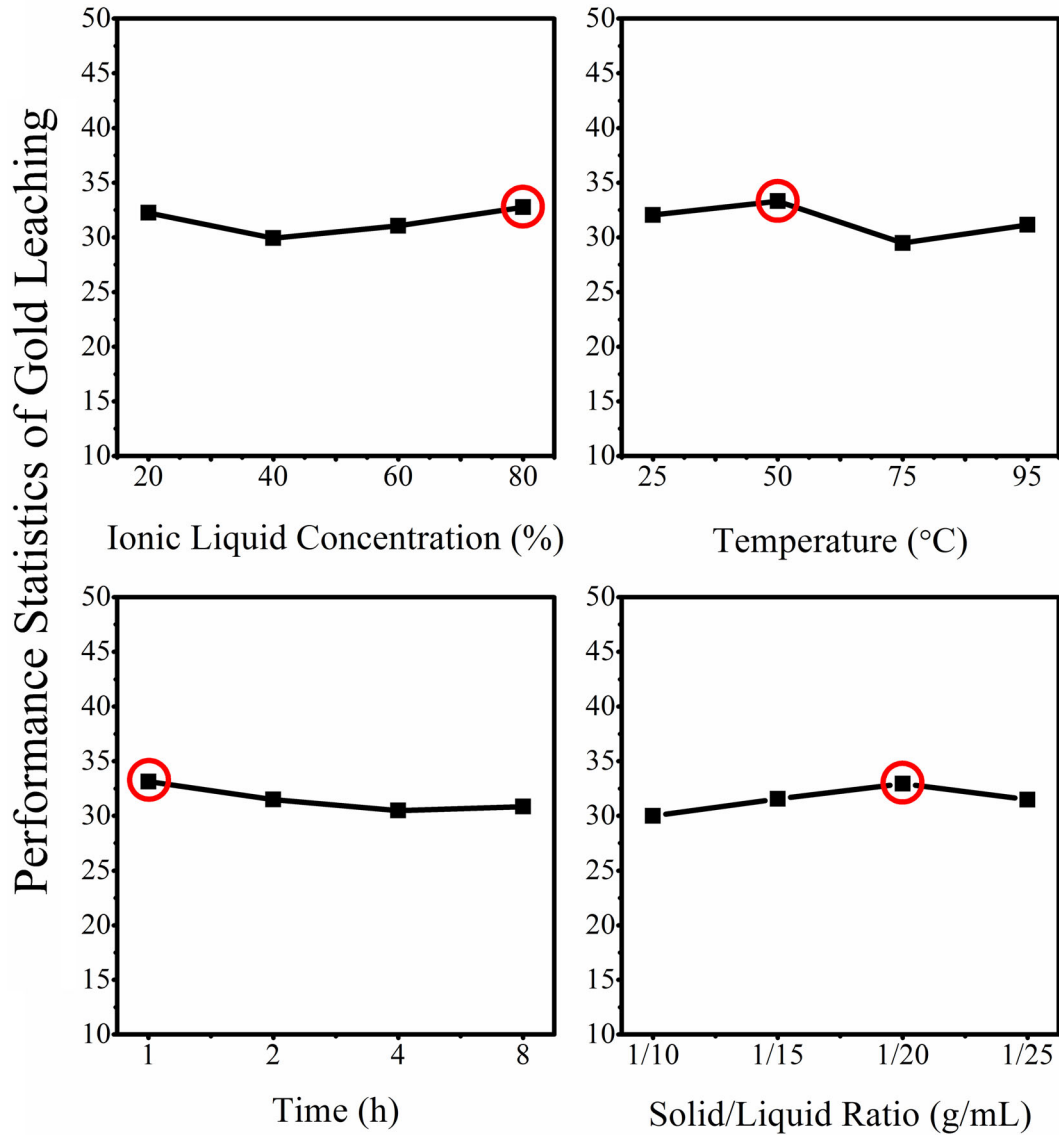


Fig. 2. Performance statistics graphs for gold leaching from CAS.

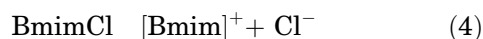
Table II. Optimum leaching conditions for gold leaching from CAS, predicted gold leaching, and results of validation experiments

Experimental leaching parameters	Optimum experimental conditions	
	Value	Level
Ionic liquid concentration	%80	4
Temperature	50°C	2
Time	1 h	1
Solid/liquid ratio	1/20 g/ml	3
Predicted gold leaching	63.3%	
Verified gold leaching	61.2%	

CAS. For this reason, the Taguchi method proposes the use of ANOVA to determine the ordering of the parameters affecting the gold leaching.^{30,31} The ANOVA results for gold leaching from CAS with BmimCl are given in Table III.

Among the investigated parameters, it was revealed that temperature was the most significant parameter for gold leaching, with a 42.5% contribution rate. In addition, the significance of the parameters for gold leaching was followed by time with 23.7%, solid/liquid ratio 17.3%, and IL concentration 16.5%. As stated in a previous study,³² it is seen that the temperature of the leaching media with Cl⁻ anions is very effective in gold leaching. Although it is known that the solubility of metals increases as the temperature increases, both the concentration of chlorine anions and gold leaching efficiency decrease due to the decomposition of BmimCl at high temperatures. Therefore, it was reported that a leaching temperature below 60°C is suitable for gold leaching by chlorination.^{32,33}

As stated by Crowhurst et al., the chemical decomposition of BmimCl in water can be given by:³⁴



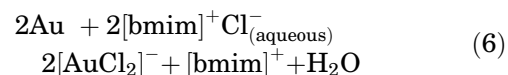
$$K = \frac{[\text{Bmim}^+][\text{Cl}^-]}{[\text{BmimCl}]} \quad (5)$$

Where K is the dissolution coefficient.

According to Eq. 5, as the concentration of the BmimCl IL increases, the number of [Cl⁻] ions increases. Hence, BmimCl provides a suitable environment for metal leaching due to the high Cl⁻ concentration and acidic properties of the IL.³⁵ As shown in Fig. 3, a stable region of the gold–chlorine complex is in place and chloride act as an oxidant that promotes gold dissolution. This stable region clarifies that the gold leaching from the CAS is feasible from the thermodynamic factors, such as the potential and pH. In the aqueous chloride system, it exists as stable under the conditions of pH below 7 and potential above 0.9 V. Conversely, as the acidity of the solution decreases, the gold

begins to precipitate in the form of Au(OH)₃. Therefore, gold can be leached in this form by chlorination when the leaching condition is acidic and has a relatively high potential. Before the leaching experiments, it was determined that the pH value and potential of an aqueous solution of BmimCl were in the range of 4–5 and above 1.2 V, respectively, which indicates a suitable leach medium for gold from the CAS.

For this reason, the leaching reaction for gold from the CAS with chlorine anions can be given as:



When the effects of the leaching parameters are examined with an orthogonal array, it may be possible that the parameters affect each other since different levels are used in each step. As indicated by ANOVA analysis (Table III), it is understood that the most effective parameters are temperature and time for gold leaching from the CAS with BmimCl. As can be seen from the limited number of studies^{18,19,21} in the literature on gold recovery with BmimCl (from gold ore and PCBs), high gold yields (up to 85%) have been obtained with the addition of various oxidants or complexing agents after relatively long leaching durations (up to 48 h). Although the 63% gold recovery by the Taguchi method in our study for very short leaching duration (1 h) without using any additives (oxidant or complexing agent) could be considered a good value, additional experiments have been carried out to observe the effects of duration on the leaching efficiency of gold from the CAS. For this reason, the effect of longer leaching times on gold leaching was investigated in the range of 1–36 h by keeping other parameters constant: 50% v/v IL concentration, 50°C temperature, and 1/20 solid/liquid ratio without any oxidant addition. The experimental results are shown in Fig. 4.

From the results in Fig. 4, it is obvious that the gold leaching efficiency increases with increasing

Table III. ANOVA results of gold leaching with BmimCl

Source	df	SS	MS	F value	Contribution%
Ionic liquid concentration	3	185.5	61.9	0.23	16.5
Temperature	3	476.0	158.6	0.59	42.5
Time	3	268.8	89.6	0.33	23.7
Solid/liquid ratio	3	195.7	65.2	0.24	17.3
Error	3	812.5	270.8		
Total	15				

df degree of freedom, SS sum of squares, MSS, mean of sum of squares.

the leaching time in BmimCl. Although the leaching yield of gold under the selected parameters was only 27.4% at the end of 1 h of leaching, after this point it dramatically and continuously increased, reaching 82.2% for 36 h of leaching. Thus, it was revealed that gold leaching with BmimCl could be improved with a longer leaching time without any additives.

Analysis of Leach Residue

Mineralogical analysis of the secondary residue obtained after the leaching experiments in BmimCl under the optimum conditions determined for gold leaching from the CAS was carried out by XRD analysis. Figure 5a indicates the comparative results of the XRD analysis between the CAS and the secondary residue obtained after the BmimCl leaching under the optimum conditions. It can be observed from Fig. 5a that the intensities of the peaks belonging to SnO_2 and BaSO_4 increased

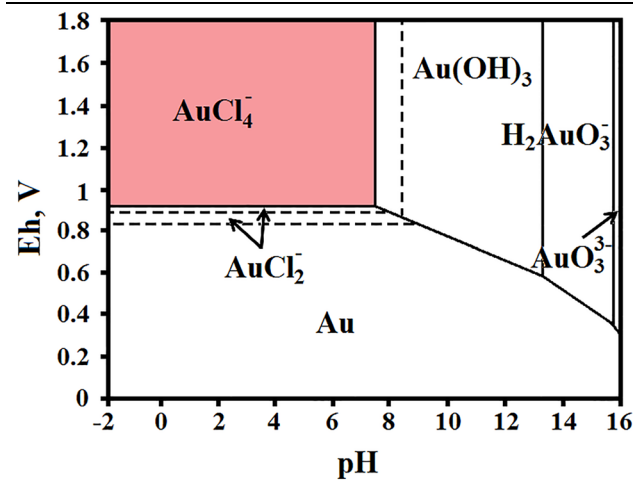


Fig. 3. The potential–pH diagram of the Au–Cl–H₂O system (reprinted with permission from Pak et al.³²).

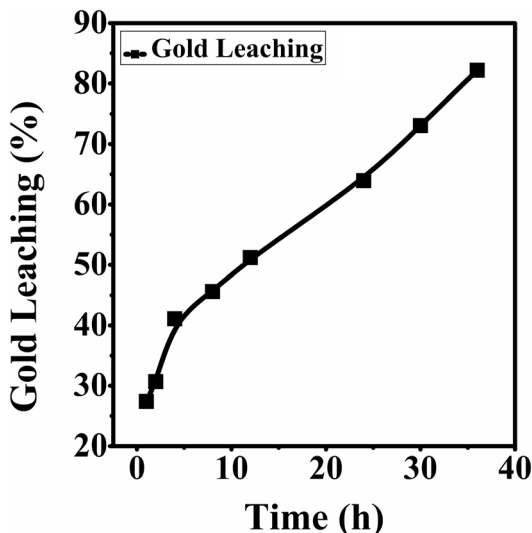


Fig. 4. Effect of time on gold leaching from the CAS with BmimCl.

highly after the BmimCl leaching, which indicates that SnO_2 and BaSO_4 were insoluble in BmimCl. In addition, the intensity of the peaks belonging to the PbSO_4 decreased after the BmimCl leaching. This refers to the slightly soluble PbSO_4 compound in BmimCl. It is known from a previous study³⁶ that the PbSO_4 structure was not soluble in an acidic leach medium like the BmimCl solution (pH: 4–5). However, it was pointed out that temperature is effective in PbSO_4 solubility in the experiments carried out in acidic conditions in the presence of Cl^- ions.³⁷ After the leaching experiments following the $\text{L}_{16}(4^4)$ experimental setup in an acidic environment at high BmimCl concentration and high temperatures (Exp. Nos: 11, 12, and 16), it was observed that fine white particles have been immediately formed in the leach solution during the filtration step. According to the SEM/EDX analysis of these white particles (Fig. 5b), it was determined that the white-colored precipitates formed after the leaching process with the BmimCl IL belong to lead sulfate (PbSO_4). This result confirms that a considerable amount of PbSO_4 can be dissolved together with gold in the BmimCl solution. These interesting facts reveal that the dissolved part of the lead can be separated from the leach solution by precipitating as lead sulfate after hot leaching.

The overall goal of this work was to investigate gold leaching from the CAS. Together with this, the leaching efficiency of other valuable elements (Cu, Ag, Se, and Te) in the CAS under the optimum conditions was determined by ICP-OES. The

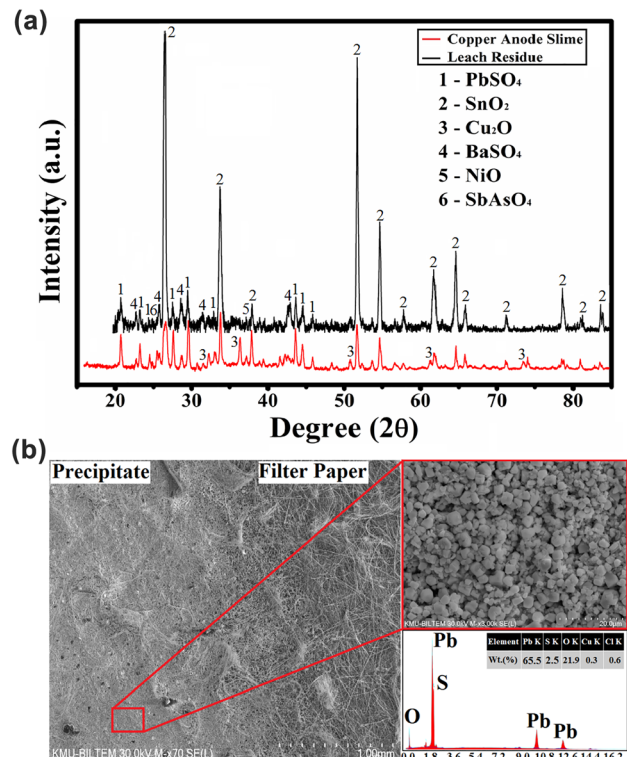


Fig. 5. (a) XRD pattern of leach residue and (b) SEM images of filter paper and white particles.

chemical composition of the final leach solution and the leaching efficiencies of other valuable elements under the optimum conditions for gold from the CAS by BmimCl are shown in Fig. 6a and b, respectively.

The results show that Cu_2O in the CAS could be leached by BmimCl. The copper leaching efficiency was obtained as 65.2% with BmimCl leaching. The solubility of the selenium and tellurium elements under the optimum conditions for gold leaching were determined as 47% and 17%, respectively. Silver could not be detected in the leach solution by ICP-OES.

As seen from Fig. 6b, the final leach solution contains Cu, Au, Se, and Te at 3765 mg/L, 0.335 mg/L, 4.85 mg/L, and 0.353 mg/L, respectively. Therefore, the precious metals should be separated from the multi-metal-containing leach solution by appropriate methods. As known from the literature, several methods can be used for gold extraction from solutions such as liquid-liquid extraction with various extractants, cementation with zinc powder, ion exchange with some resins, and adsorption of gold with active carbon.³⁸⁻⁴¹ Among them, liquid-liquid extraction is generally used for the purification of metals from multi-element leach solutions.⁴²⁻⁴⁴ Extensive studies have been conducted by several researchers to develop efficient processes for the separation and purification of precious metals from various leach liquors using solvent extraction (SX). A flowchart for the separation and recovery of the noble and base metals in anode slime from the chloride leaching solution by using solvent extraction and stripping methods has been proposed by Xing et al.⁴⁵ According to this study, if gold is present in the leach solutions with Cu and Se, it is usually the first metal to be extracted by using an organic extraction solvent such as Cyanex 272.

After that, Cu could be obtained by SX with LIX 63 and stripping with HCl solution. Finally, it has been proposed that Ag (not in our leach liquor) in the solution could be extracted with Alamine 336 and stripped with NH_4SCN . Although there are such case studies for gold recovery from chloride leach solutions, further investigation should be performed for gold extraction from the BmimCl leach solution.

As stated in the previous studies,⁴⁶⁻⁴⁸ ILs based on imidazolium cations can be recycled and reused several times without any change in their structure and impurity by using methods such as distillation, extraction, absorption, etc. It has been recently reported that BmimCl IL can be recovered and recycled without any problems due to the degree of the solution value being very low even after five cycles.⁴⁹ The industrial usability of ILs will increase with the decrease in their production costs and the increase in the number of leach cycles. Therefore, it is expected that BmimCl used in the leaching process can be reused for the metal extraction process without polluting the waste water and environment in the future.

CONCLUSION

The Taguchi optimization method has been used for determining the optimum leaching conditions for gold leaching from the CAS in the BmimCl IL medium. Leaching parameters were investigated with an orthogonal experimental design. To obtain the highest gold leaching efficiency from the CAS by BmimCl, the optimum leaching conditions were selected as: 80% IL concentration, 50°C temperature, 1 h leaching time, and 1/20 solid/liquid ratio. Under these conditions, gold leaching efficiencies showed an excellent relationship between the

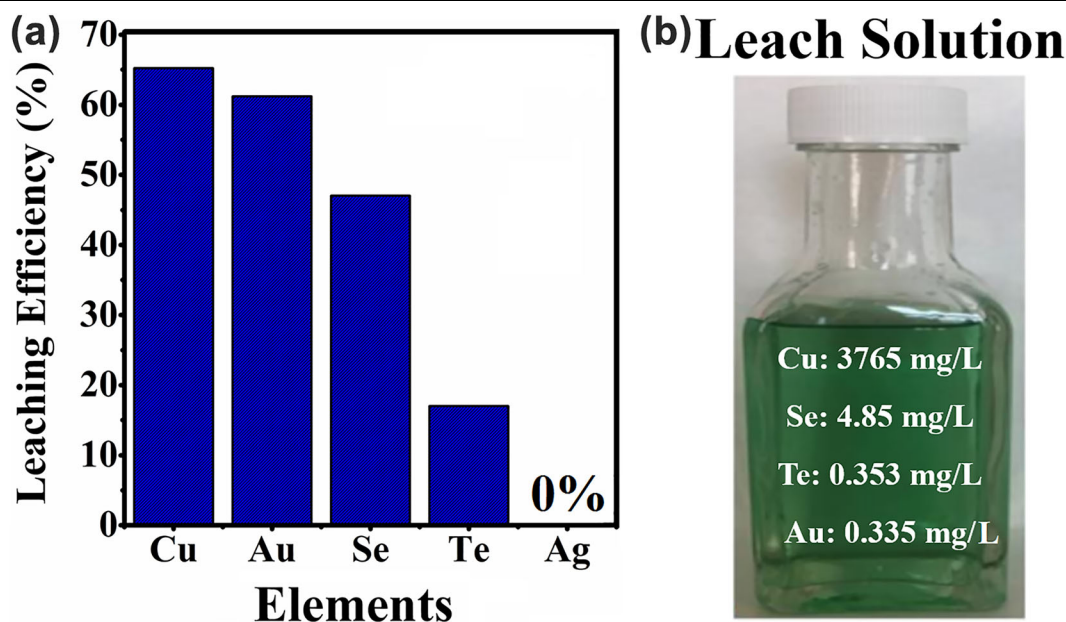


Fig. 6. (a) Leaching efficiency of valuable elements by BmimCl and (b) chemical composition of the final leach solution.

validation experiment (63.3%) and the theoretical value (61.2%). ANOVA revealed that the most effective parameter in gold leaching was the temperature, at 42.5%. As a result of this study, it is thought that the hydrometallurgical process in the presence of BmimCl can be successfully applied for gold leaching from the CAS.

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CONFLICT OF INTEREST

The authors declare that they have no conflict of interest.

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