Development of a hydrometallurgical process for the recovery of gold and silver powders from anode slime containing copper, nickel, tin, and zinc

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Abstract
In order to recover gold and silver from anode slimes containing Cu, Ni, Sn, and Zn, an integrated hydrometallurgical process consisting of leaching, solvent extraction, and cementation was developed. All the metals together with 10% of Ag(I) were dissolved by the mixture of HCl and H 2O2 at the optimum conditions. Separation of Au (III) together with Sn (II) was performed by Cyanex 272 from the leaching solution with two-stage counter current extraction. Stripping of Au (III) and Sn (II) from the loaded organic phase was sequentially carried out by NH 4Cl and NaOH. Cu (II) in the raffinate after Au (III) extraction was separated by LIX 63 with three-stage counter current extraction. Pure Cu (II) solution was recovered from the loaded LIX 63 by stripping with dilute HCl solution. Ag powders with extra high purity were obtained by cementation with copper sheet from the raffinate after Cu (II) separation. Au (III) (99.3%) and 96.8% of Sn (II) were recovered by extraction, and purity of Au (III) and Sn (II) stripping solutions was found to be 99.99%. Au powders with extra high purity were directly synthesized by adding ascorbic acid solution into the NH 4Cl stripping solution.

Keywords Copper anode slimes · Solvent extraction · Gold powders · Silver powders · Cementation

Introduction
The demand for gold and silver becomes increasing because these metals are raw materials for the manufacture of advanced materials for jewelry, electrical, electronic, and medical industry [1]. Therefore, it is essential to recycle these metals from the secondary resources [1–3]. Among the secondary resources, anode slimes are generated from the electrorefining of metals and contain Au, Ag, and platinum group metals (PGMs). Various environment-friendly technologies have been developed for the recovery of the valuable metals from anode slimes.

In general, combination of hydrometallurgical and pyrometallurgical processes is extensively practiced for the enrichment of the anode slime and subsequent dissolution of the enriched slimes. In conventional recovery processes for different kinds of anode slimes, copper was firstly separated by several methods like oxidizing roasting [4], concentrated sulfuric acid solution leaching [5], and pressure leaching [6]. Meanwhile, noble metals present in anode slimes are enriched by selective dissolution of base metals [7, 8]. Then, the enriched noble metals can be dissolved in various reagents such as halide, thiourea, and thiosulfate in the presence of oxidizing reagents [5, 9–12]. Some methods such as cementation, adsorption, solvent extraction, and ion exchange are employed for the separation of gold, silver, and PGMs from the leaching solution [13–17].

Some works have proposed integrated processes for the recovery of valuable metals from anode slimes by combing pyro- and hydrometallurgical processes [2, 5, 7, 18]. Among these methods, pretreatment was generally employed by either alkali fusion leaching or sulfuric acid leaching for the removal of some metals such as Cu, As, Te, and Sn. Then, noble metals were enriched by dissolving impurities with various reagents. The disadvantage of these processes is high consumption of energy and alkali fusion roasting results in more hazardous wastes and corrosion atmosphere. Moreover, multi-stage leaching brings about the loss of noble metals and the production of acidic waste effluents. In our previous work, we have
reported the separation of valuable metals from anode slimes by hydrometallurgical methods consisting of leaching [9], solvent extraction [19–21], ion exchange [22], and cementation [23]. Without any pretreatment steps for the enrichment of precious metals from the anode slimes, employment of the mixture of HCl and H2O2 was efficient for the dissolution of all the metals except Ag [9]. Sequential separation of gold, tin, and copper was done from the synthetic leaching liquor by extraction with Cyanex 272 and LIX 63, respectively [19, 20]. Among some separation methods like solvent extraction, ion exchange, and cementation, cementation by copper was found to be the most efficient method for silver recovery [20, 23]. Although recovery of gold and silver was successfully performed by those methods from the synthetic HCl solution, the mass balance of the elements in the anode slimes was not reported in our previous work.

In order to verify the process for the recovery of Au and Ag present in the anode slimes, real leaching solutions of anode slimes were employed in this work. In each step consisted of leaching and separation of the metals by solvent extraction, the optimum conditions obtained from the previous works were employed. Namely, the anode slimes containing Ag, Au, Cu, Ni, Sn, and Zn were dissolved by the mixture of hydrochloric acid and H2O2. Au (III) was first separated by solvent extraction with Cyanex 272 and then Cu (II) was separated by solvent extraction with LIX 63. For each solvent extraction, batch simulation extraction and stripping experiments were done on the basis of the corresponding McCabe-Thiele diagrams. The purity and recovery percentage of Au (III), Ag(I), and Cu (II) were obtained. Moreover, the powders of Au and Ag were synthesized from the recovered solutions.

**Materials and methods**

**Materials and analysis**

The composition of the anode slimes supplied by a Korea company is represented in Table 1. The materials were dried in an oven for several days and then milled into 100-μm particle size. The characteristics of the anode slime was investigated by X-ray diffraction (XRD, D8 Advance, Bruker AXS, USA), analytical high-resolution scanning electron microscopy (HR-SEM, SU-70, Hitachi, Japan), and energy-dispersive spectroscopy (EDS, EDAX STD-S, No. PV7747/67). The XRD pattern of the anode slimes is shown in Fig. 1. According to the XRD data, the primary phases in the anode slimes were found to be Cu2O·(SO4)·(OH)4, CuSO4·5H2O, Cu2O along with metallic Cu. Meanwhile, gold and silver were present as metallic state. SEM image and EDS spectrum of anode slime are shown in Fig. 2, which confirmed the existence of Sn and low content of Ni and Zn in the anode slime. The extractants used in this work like Cyanex 272 (85%, Cytec Solvay Industries, USA) and LIX 63 (70%, BASF Co., USA) were diluted by kerosene (Dae Jung Chemical Co., Korea). All extractants were employed as received from the suppliers without any treatment. Kerosene was used for the diluent. All the chemicals used in experiments were of analytical grade.

**Experimental procedures**

The leaching solution of anode slimes was obtained by directly dissolving the anode slimes with the mixture of HCl and H2O2 in the optimum conditions of 5 M HCl and 0.2 M H2O2, temperature of 90 °C, 100 μm particle size, 2 h reaction time, pulp density of 10 g/L, and stirring speed 200 min⁻¹. Solvent extraction experiments were performed by mixing equal volume of organic and aqueous phase in a 50-mL screw bottle, and the mixtures were shaken within 30 min with a wrist action shaker (Burrell, Model 75) at room temperature (25 ± 1 °C). Batch simulation extraction experiments were done in a 500-ml beaker by mixing equal volume of aqueous and organic phase at the stirring speed of 300 rpm for 30 min at room temperature. After shaking, separation of organic and aqueous phase was carried out in the separation funnel. The concentration of metal ions in the aqueous phase was measured by ICP-OES (inductively coupled plasma optical emission spectroscopy, Spectro Arcos, Cleve, Germany). The corresponding

![Fig. 1 X-ray diffraction patterns of the copper anode slimes supported by Korean company](image_url)

| Cu  | Zn  | Ni  | Ag  | Sn  | Au  |
|-----|-----|-----|-----|-----|-----|
| Contents, wt% | 41.1 | 0.200 | 0.300 | 4.60 | 5.80 | 1.50 |
| Leach liquor, mg/L | 1752 | 9.10 | 15.5 | 39.7 | 326 | 234 |

[HCl] = 5 M, [H2O2] = 0.2 M, stirring speed 200 min⁻¹, pulp density 10 g/L, reaction time 2 h, temperature 90 ± 2 °C
concentration of metal ions in the organic phase was obtained by mass balance.

**Gold recovery** The experiments were done in a 200-ml beaker on the heater with a magnetic stirrer. Fifty milliliters of NH₄Cl stripping solution was mixed with ascorbic acid (99.5%, Samchun Pure Chemical Co., Ltd., Korea). The stirring speed was controlled at 300 rpm. When the color of the solution changed into purple red, the solution was kept for 30 min. The gold particles thus obtained were analyzed by FE-SEM (S-3500 N Hitachi, Tokyo, Japan).

**Ag recovery** After Cu extraction by LIX 63, most of silver remained in the raffinate. Silver cementation was performed by copper sheet in a 200-mL beaker with the stirring speed of 200 rpm. When the cementation was completed, the silver thus obtained on the copper sheet was washed by distilled water. The concentration of metals in the solution after cementation was measured by ICP-OES.

### Results and discussion

#### Leaching of the anode slimes

In the leaching of the anode slimes with the mixture of hydrochloric acid and H₂O₂, most of the metals in anode slime except Ag was completely dissolved [9]. Therefore, the real leaching solution was obtained at the optimum condition; 5 M HCl and 0.2 M H₂O₂, reaction temperature of 90 °C, 100 μm particle size, reaction time of 2 h, pulp density of 10 g/L, and stirring speed 200 min⁻¹. Under these conditions, more than 99.99% of Au, Cu, Zn, Ni, and Zn as well as 10% of Ag were dissolved in the HCl solution and the metal concentration in the leaching solution of anode slimes is shown in Table 1.

Gold exists as AuCl₄⁻ in the leaching solution, while silver exists as either AgCl₂⁻ or AgCl₃⁻. Since all the other metals were dissolved in the HCl solution, the precipitated silver was directly recovered as the form of AgCl.

#### Gold recovery

In previous work, Au (III) was completely separated by solvent extraction with Cyanex 272 from the synthetic leaching solution. The extraction reaction of Au (III) with Cyanex 272 can be represented as Eq. (1) [19].

\[
\text{HAuCl}_4^- + H^+ + 2\text{HA}_{org} = [\text{HAuCl}_4 \cdot 2\text{HA}]_{org} \quad (1)
\]

where HA refers to Cyanex 272.

#### Gold extraction

In order to confirm the complete separation of Au (III) from the real leaching solution of anode slimes, extraction experiments were carried out by varying Cyanex 272 concentration from 0.01 to 0.25 M and the results are shown in Fig. 3. As Cyanex 272 concentration increased from 0.01 M to 0.05 M, the extraction of Sn (II) increased rapidly from 20% to 90% and then remained at 95% with the further increase of Cyanex 272 concentration. By contrast, the extraction of gold (III) increased slowly to 95% with the increase of Cyanex 272 concentration. In these experiments, other metals except Au (III) and Sn (II) were not extracted at all, which agreed well with the previous data [19].

Cyanex 272 (0.2 M) was selected for the batch simulation extraction experiments. Figure 4 shows the results on the batch simulation of two-stage counter-current extraction of Au (III). The concentration and the extraction percentage of the metals in the corresponding raffinate are displayed in Table 2. After two-stage counter current extraction, the extraction percentage of Au (III) and Sn (II) was 99.3% and 96.2%,

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**Fig. 2** SEM image and EDS spectrum of anode slimes used in this work
respectively. Moreover, Ag(I), Cu (II), Ni (II), and Zn (II) were not extracted at all during the batch simulation experiments.

**Gold stripping**

After batch simulation experiment of two-stage counter current extraction, the concentration of Au (III) and Sn (II) in the loaded Cyanex 272 was 232.8 mg/L and 315.8 mg/L, respectively. According to the previous work, (NH4)2S2O3, (NH2)2CS and NH4Cl can completely strip the Au (III) from the loaded Cyanex 272 [19]. Among these three stripping reagents, NH4Cl showed better performance in terms of stripping percentage and separation factor. Therefore, NH4Cl was employed in this work and the effect of NH4Cl concentration is shown in Fig. 5. Only Au (III) was stripped from the loaded Cyanex 272 and the stripping percentage of Au (III) increased linearly as NH4Cl concentration increased from 0.1 M to 0.5 M, and about 92% of Au (III) was stripped from the loaded organic phase. Since no Sn (II) was stripped into the NH4Cl solution, the purity of Au (III) in NH4Cl solution was higher than 99.99% and this solution is going to be employed for the preparation of gold particles in subsequent process.

**Preparation of gold powders**

Many literatures have reported that gold particles can be synthesized by adding ascorbic acid [24], sodium citrate [25], and NaNH4 [26] to the HCl solution. The reduction reaction of Au (III) by ascorbic acid can be represented as Eq. (2) [27]. In this work, ascorbic acid of $6 \times 10^{-3}$ M was added to the NH4Cl stripping solution where the concentration of Au (III) was 172.6 mg/L and the temperature was kept at 30 °C.

$$2\text{AuCl}_4^- + 3\text{C}_6\text{H}_8\text{O}_6 = 2\text{Au} + 8\text{Cl}^- + 3\text{C}_6\text{H}_6\text{O}_6 + 6\text{H}^+ \quad (2)$$

The SEM image of the gold powders thus obtained is shown in Fig. 6. The morphology of most of gold particles...
was sphere, and the particle with 1 μm size was equally distributed.

**Tin recovery**

After stripping of gold (III) from the loaded Cyanex 272, most of Sn (II) remained in the organic phase. Therefore, NaOH solution was employed for the stripping of Sn (II) from the loaded Cyanex 272. Figure 7 shows the effect of NaOH concentration on the stripping of Sn (II). The concentration of Sn (II) in the loaded Cyanex 272 was 315.8 mg/L and 0.5 M NaOH led to stripping percentage of 99.9% (see Fig. 7). The slight decrease in the stripping percentage of Sn (II) at 2.5 M NaOH solution might be ascribed to the formation of emulsion at this stripping condition [28]. Therefore, 1 M NaOH solution was selected for the optimum condition for Sn recovery.

**Copper recovery**

**Copper extraction**

Cu (II) can be selectively extracted by LIX 63 from the synthetic raffinate after Au (III) extraction [20]. The composition of the raffinate after the extraction with Cyanex 272 is shown in Table 2. Comparison between Tables 1 and 2 reveals that there was no change in the concentration of the metal ions except Au (III) and Sn (II). Figure 8 shows the effect of LIX 63 concentration on the extraction of the metal ions in the raffinate. The extraction percentage of Cu (II) increased with increasing LIX 63 concentration, and the increased rate became slow from 0.15 M LIX 63 and the extraction percentage of Cu (II) was around 90% at 0.2 M LIX 63. Extraction of Zn (II), Ni (II), and Sn (II) increased slowly to 48%, 35%, and 33% as LIX 63 concentration increased from 0.01 to 0.25 M, respectively. However, no Ag(I) was extracted into LIX 63 within the experimental ranges.

Since not only Cu (II) but also Ni (II) and Zn (II) were extracted into LIX 63, selection of a suitable concentration of LIX 63 is very important in separating Cu (II) from the raffinate. LIX 63 (0.2 M) was selected in this work, and batch simulation of three-stage counter current extraction was performed at a phase volume ratio of unity. Figure 9 shows the detailed procedure for the batch simulation experiments and the corresponding concentration, and the extraction percentage of the metals are shown in Table 3. Ninety-
eight percent of Cu (II) together with 52% of Ni (II) was extracted into LIX 63, while most of the Ag(I) remained in the raffinate.

**Copper stripping**

According to the previous work, HCl showed good performance as a stripping agent for the Cu (II) in the loaded LIX 63 [20]. After the batch simulation of three-stage counter current extraction, the concentration of Cu (II) and Ni (II) in the loaded LIX 63 was 1718 mg/L and 9.1 mg/L, respectively. Figure 10 shows the effect of HCl concentration on the stripping of Cu (II) and Ni (II) from the loaded LIX 63. There was not much increase in the stripping percentage of Cu (II) and Ni (II) as HCl concentration increased from 0.001 M to 0.025 M. When 0.015 M HCl was employed, the stripping percentage of Cu (II) and Ni (II) was 80% and 20%, respectively. The purity of Cu (II) in the stripping solution was 99.8% at 0.015 M HCl.

**Silver recovery**

After most of Cu (II) was separated from the raffinate by extraction with LIX 63, almost 40 mg/L of Ag(I) remained in the raffinate. Ion exchange and solvent extraction were not effective in separating Ag(I) from the raffinate [20, 23]. However, cementation of Ag(I) by copper metal was found to be effective for the selective recovery of silver metal from the raffinate [23]. The reactions which might occur during the cementation are represented in Eqs. (3) and (4). Therefore, copper sheet was employed for the recovery of silver metal from the raffinate (C3) in this work (see Table 4). The cementation conditions were temperature of 90 °C, reaction time of 60 min, stirring speed of 200 min⁻¹, and 50 × 25 × 1 mm³ copper sheet. Table 4 shows that the recovery percentage of Ag(I) was 97%. Moreover, there was no change in the concentration of other metal ions in the raffinate except Ag(I), indicating that only Ag(I) was cemented by copper sheet from the raffinate and the purity of Ag metal might be higher than 99.9%. Figure 11 shows the SEM image of silver powders thus obtained. The size of the particles was less than 0.5 μm and the morphology looked like cubic:

![Fig. 9 Recovery of Cu(II) by three-stage counter current extraction with LIX 63 from the raffinate after extraction of Au (III) and Sn(II). [LIX 63] = 0.2 M, O/A = 1](image)

**Table 3** Extraction of Cu by LIX 63 with three stage counter current extraction

|        | Cu   | Zn  | Ni  | Ag  | Sn  |
|--------|------|-----|-----|-----|-----|
| Raffinate, mg/L | 1751 | 9.60| 16.6| 37.9| 10.3|
| Stage 1 Extraction % | 98.1 | 0   | 54.2| 1.50| 0   |
| A3 Raffinate, mg/L | 33.3 | 9.90| 7.60| 37.3| 10.5|
| Stage 2 Extraction % | 98.1 | 0   | 52.5| 0   | 0   |
| B3 Raffinate, mg/L | 33.8 | 9.30| 7.90| 41.3| 12.5|
| Stage 3 Extraction % | 97.9 | 0   | 54.6| 0   | 0   |
| C3 Raffinate, mg/L | 36.7 | 9.80| 7.50| 38.5| 14.5|

[LIX 63] = 0.2 M, O/A = 1

![Fig. 10 Effect of HCl concentration on the stripping of Cu(II) from the loaded LIX 63. In loaded LIX 63: [Cu(II)] = 1718 mg/L, [Ni(II)] = 9.1 mg/L; O/A = 1](image)

**Table 4** The concentration of metals in the solution before and after Ag cementation by copper sheet

| Contents          | Cu   | Zn  | Ni  | Ag  | Sn  |
|-------------------|------|-----|-----|-----|-----|
| Raffinate C3, mg/L | 35.8 | 6.90| 7.50| 38.8| 10.4|
| After Ag cementation, mg/L | 365 | 7.10| 7.50| 1.20| 10.5|
| Recovery %        | 0   | 0   | 96.9| 0   | 0   |

Temperature 90 °C, reaction time 60 min, stirring speed 200 min⁻¹, 50 × 25 × 1 mm³ copper sheet
2AgCl₂⁻ + Cu(s) = CuCl₄²⁻ + 2Ag(s)  \hspace{1cm} (3)

Cu(s) + CuCl₄²⁻ = 2CuCl₂⁻  \hspace{1cm} (4)

Proposal of a process

Figure 12 shows the hydrometallurgical flowsheet proposed in this work for the production of extra pure gold and silver particles from the anode slimes. Table 5 shows the mass balance and the purity of metals present in the anode slime in each extraction and stripping step as well as cementation. All the metals except silver in anode slime were completely dissolved by the mixture of hydrochloride acid and H₂O₂. Au (III) and most of Sn (II) were selectively separated by Cyanex 272 with two-stage counter current extraction from the leaching solution of anode slime. Pure Au (III) solution was recovered by stripping of the loaded Cyanex 272 with NH₄Cl solution and then the remaining Sn (II) can be stripped by NaOH solution. Extra pure gold particles can be synthesized by adding ascorbic acid to the NH₄Cl stripping solution. Most of Cu (II) together with a small amount of Ni (II) in the raffinate was extracted by LIX 63 with three-stage counter current extraction. Pure Cu (II) solution was recovered by stripping with dilute HCl solution. The remaining silver in the raffinate after Cu (II) extraction was recovered by cementation with copper sheet, while the dissolved copper could be recovered by extraction with LIX 63.

Compared to other processes proposed for the recovery of metals from anode slimes, the process proposed in this work do not need any pretreatment step for the enrichment of noble metals in the anode slime. Moreover, pure Au (III) and Cu (II) solutions were obtained from the leaching solution by extraction and stripping. Gold particles can be obtained from the stripping solution.
Conclusions

A hydrometallurgical process has been developed for the recovery of pure gold and silver particles from the anode slime containing Cu, Ni, Sn, and Zn. Above 99.9% of Au, Cu, Ni, Sn, and Zn and 10% of Ag in the anode slime were dissolved by the mixture of HCl and H₂O₂ at the optimum conditions. Separation of Au (III) was performed by extraction with Cyanex 272 and stripping with NH₄Cl from the leaching solution of anode slimes. The Sn (II) remaining in the stripped Cyanex 272 was successfully stripped by using NaOH solution. Cu (II) together with a small amount of Ni (II) in the raffinate after Au (III) extraction was selectively separated by LIX 63 from which Cu (II) was stripped by dilute HCl solution. After batch simulation extraction experiments, gold particles with 99.99% purity were prepared by reduction with ascorbic acid. Silver powders with extra purity were recovered by cementation with copper sheet from the raffinate after the extraction of Cu (II). The purity of Cu (II) and Sn (II) in the respective stripping solutions was found to be 99.8% and 99.99%, respectively. The overall recovery of gold and silver from anode slime was 91.4% and 96.9%.

The anode slime employed in this work contained six metals. By applying the process developed in this work, extra pure Ag and Au particles can be obtained from the leaching solution. Moreover, pure Cu (II) and Sn (II) solutions can be recovered by this process. The fact that this process does not need any pretreatment step for the enrichment of the anode slimes is one of the advantages of this process. However, some more work need to be done for the disposal of the effluent after the cementation of Ag(I) by copper.

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Table 5 Mass balance of metals in each step of the process developed in this work

| Process         | Detail                          | Au   | Ag   | Cu   | Sn   | Ni   | Zn   |
|-----------------|---------------------------------|------|------|------|------|------|------|
| Raw material    | Anode slimes, wt%               | 1.50 | 4.60 | 41.1 | 5.80 | 0.300| 0.200|
| Leaching        | Leaching solution, mg/L         | 234  | 39.7 | 1752 | 326  | 15.5 | 9.10 |
|                 | Leaching %                      | 99.9 | 10   | 99.9 | 99.9 | 99.9 | 99.9 |
| Au recovery     | Solution after extraction, mg/L | 1.60 | 40.2 | 1752 | 12.5 | 16.0 | 9.30 |
|                 | Aqueous phase after stripping, mg/L | 173  | 0    | 0    | 0    | 0    | 0    |
| Sn recovery     | Solution after extraction, mg/L | 1.60 | 40.2 | 1752 | 12.5 | 16.0 | 9.30 |
|                 | Aqueous phase after stripping, mg/L | /    | 38.5 | 36.7 | 14.5 | 7.50 | 9.80 |
| Cu recovery     | Solution after extraction, mg/L | /    | 1.20 | 365  | 10.5 | 7.50 | 9.10 |
|                 | Aqueous phase after stripping, mg/L | /    | /    | 1374 | /    | 1.80 | /    |
| Ag recovery     | Aqueous after Ag cementation, mg/L | /    | /    | 98.1 | 96.8 | /    | /    |
| Results         | Extraction percentage, %        | 99.3 | 92.0 | 91.4 | 91.4 | 91.4 | 91.4 |
|                 | Stripping percentage, %         | /    | 98.0 | 99.9 | 99.9 | /    | /    |
|                 | Recovery percentage, %          | /    | /    | 80.0 | 80.0 | /    | /    |
|                 | Purity, %                       | 99.9 | 99.9 | 99.8 | 99.8 | 99.9 | 99.9 |

Compliance with ethical standards

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