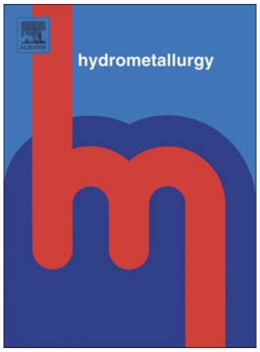

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Novel process for recycling gold from secondary sources: Leaching of gold by dimethyl sulfoxide solutions containing copper bromide and precipitation with water

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Keywords: Dimethyl sulfoxide, copper bromide, gold dissolution, precipitation, waste of electric and electronic equipment, water

ABSTRACT

A novel method for recovering gold from secondary sources, the leaching of gold using dimethyl sulfoxide (DMSO) solutions containing copper bromide followed by the precipitation of gold with water, is presented. Gold dissolution was conducted in a DMSO solution with 0.1–0.2 M $CuBr_2$ and 0–0.2 M KBr at 333–348 K. The mechanism of gold dissolution was investigated by electrochemical measurements. The precipitation of dissolved gold was performed by the addition of water, during which the effects of the amount of water and the pH on the recovery rate were investigated. It was found that the initial gold dissolution rate in the DMSO solutions with $CuBr_2$ was up to 37 mg cm^{-2} h^{-1}, which is larger than those obtained with other leaching methods. The results of the electrochemical measurements indicate that the anodic dissolution of gold in DMSO containing $CuBr_2$ occurs at relatively negative potentials and is paired with the cathodic reduction of Cu^{2+} to Cu^{+}. A recovery rate of gold up to 87% was obtained by precipitation with water. A small amount of copper was precipitated with gold, which can be avoided by using water of lower pH, as is expected by the Eh-pH diagram. Our results demonstrate that a circulating system for gold leaching and recovery, which would offer a number of advantages, including eco-friendliness, easy operation, low costs, and minimization of chemical sludge production, can be developed.

1. Introduction

The recovery of gold from secondary sources such as waste electric and electronic equipment (WEEE) has received much attention because of the many industrial applications and high market prices of this extensively used precious metal (Owens, 2013; Syde, 2013). To date, various recovery techniques have been developed to recover gold from secondary sources, e.g., pyrometallurgy, hydrometallurgy and bio-hydrometallurgy techniques. Among these methods, hydrometallurgy, which uses cyanide, halide and thiourea, among other substances, as leaching agents, is now the most widespread technology (Syde, 2013). With their relatively low capital cost, reduced environmental impact (e.g., no hazardous gases/dusts), potential for high metal recoveries and suitability for small-scale applications, hydrometallurgical processes are recognized as promising options for the treatment of secondary sources (Yazici and Deveci, 2009; Tuncuk, 2012). However, restrictions on waste disposal, safety concerns and stringent environmental regulations have motivated the further development of economically viable technologies that pose fewer health risks and are more eco-friendly (Syde, 2006).

Although the leaching and recovery of gold in aqueous solutions have been extensively investigated (Marsden and House, 2006), only a few preliminary investigations on nonaqueous solutions have been reported. Nakao and Sone (1996) observed unique behavior for gold in iodine-iodide-acetonitrile solution, i.e., that gold can be dissolved upon heating and deposited upon cooling quite reversibly in solution. This system offers the advantage of the repeated use of the leaching solvent to avoid large reagent consumption and chemical sludge production. Mortier et al. (2005) used chloroform solutions of cetyltrimethylammonium bromide (CTAB) for gold oxidation and showed that solid gold is effectively oxidized in a concentrated chloroform solution of CTAB with the aid of sonication, in which Br^- from CTAB acts as a nucleophile and oxygen in the air is responsible for the oxidation of bulk gold. Mortier et al. also showed that the addition of hydrazine, a reducing agent, to the sonicated solution leads to the formation of gold nanoparticles dispersed in chloroform. Lin et al. (2010) showed that mixtures of thionyl chloride ($SOCl_2$) and certain organic solvents/reagents (pyridine, N,N-dimethylformamide and imidazole) can dissolve noble metals, including gold, at high dissolution rates under mild conditions, naming these mixtures “organic liquor regius”. Their organic liquor regius showed selectivity in noble metal dissolution. Lin et al. also showed that the dissolved gold can be recovered by calcination.

Although all of these previous investigations demonstrated some advantages of nonaqueous solutions for gold leaching and recovery, the use of solvents with high toxicity and flammability due to their high vapor pressure discourages their industrial applications for safety reasons. In addition, the consumption of relatively high-cost solvents and the production of chemical sludge remain issues to be resolved. To overcome these issues, the following are required for nonaqueous solutions to be successfully used in gold leaching and recovery: 1) low toxicity, 2) stability, 3) high rate of dissolution and recovery of gold and 4) recyclability.

In this paper, we present a novel method for recovering gold: the leaching of gold by dimethyl sulfoxide (DMSO) solutions containing copper bromide followed by the precipitation of gold with water.

DMSO is an aprotic polar solvent that is widely used in chemical industries. DMSO has a number of advantages for solvent use, including the ability to dissolve many inorganic salts, low toxicity, low vapor pressure at ambient temperature and resistance to oxidation and reduction (Butler, 1967; Zhu et al., 2013). In addition, the high redox potentials of gold can be significantly lowered in DMSO in the presence of halogens and corresponding halides (Benari et al., 1983). Additionally, both copper (I) and copper (II) ions are known to be moderately solvated in DMSO (Chaudhry, 1994). Therefore, copper (II) ions in DMSO are expected to act as an oxidizing agent and be reduced to copper (I) ions by oxidizing gold in the presence of halide ions. In addition, DMSO solvates water in any concentration. The addition of water in DMSO solutions will drastically change the solution characteristics, in particular resulting in high redox potentials of gold in mixed solutions. This behavior indicates that water can be used for the precipitation of gold. Furthermore, DMSO does not form an azeotrope with water and can be separated by distillation, which enables the repeated use of the solutions.

In this paper, the observations related to our proposed method are summarized as follows. First, electrochemical measurements of gold oxidation and copper ion reduction in the solution were conducted. Second, the effects of the concentrations of $CuBr_2$ and KBr in DMSO and temperature on the dissolution rate of gold were investigated. Third, the precipitation of dissolved gold was performed by the addition of water, during which the effects of the amount of water and pH on the recovery rate of gold and the size of the precipitated gold particles were investigated.

2. Materials and Methods

2.1 Materials

KBr (99%), tetra-n-butylammonium tetrafluoroborate (96%), DMSO (99%) and sulfuric acid (95%) were purchased from Kanto Chemical Co. Inc., Tokyo, Japan. $CuBr_2$ (99%) was purchased from Nacalai Tesque, Inc., Kyoto, Japan. Commercially available distilled water was purchased from Daiwa Ltd., Tokyo, Japan.

2.2 Methods

2.2.1 Electrochemical measurement

Steady-state polarization and cyclic voltammetry were used to measure the anodic dissolution of gold and redox potential of copper (I) and copper (II) ions in DMSO solutions, respectively. A conventional single-compartment electrochemical cell was used. The cell consisted of a gold rod (99.95%, ϕ = 1.0 mm and a length of 10 mm exposed in the solution) as the working electrode for steady-state polarization, a glassy carbon disk (ϕ = 3 mm, ALS Co., Ltd.) as the working electrode for cyclic voltammetry, a Ag/Ag+ reference electrode (ALS Co., Ltd., RE-7 for organic solvent) and a platinum rod as the counter electrode. All potentials reported in this paper are with respect to the Ag/Ag+ reference electrode scale. A potentiostat (Hokuto Denko, HA-151) and a function generator (Hokuto Denko, HB-104) were used to set and control the potentials, and current-potential curves were recorded by a data logger (Graphtec, GL220).

All of the electrochemical experiments were carried out at 343 K. For the steady-state polarization measurements, DMSO solutions containing 0.1-0.2 M $CuBr_2$ and 0.1 M KBr were used. For the cyclic voltammetry measurements, DMSO solutions containing 0.01 M $CuBr_2$ and 0.4 M tetra-n-butylammonium tetrafluoroborate as a supporting electrolyte were used. The sweep rate was set to 50 and 100 mV/s, and all scans were started at the corresponding rest potential.

2.2.2 Gold dissolution experiments

One to two millimoles each of $CuBr_2$ and KBr was dissolved in 10 ml of DMSO in a conical flask and maintained at 333–348 K. Then, 235 mg (1.2 mmol) of gold (99.95%) wire with a diameter of 0.2 mm and a length of 400 mm was placed in the solvent for dissolution on a shaking apparatus. The remaining gold wire was removed from the apparatus at various time intervals (0.5–4 h) so that its weight could be measured to calculate the amount of dissolved gold. Experiments were conducted with CuBr in argon-purged flasks to investigate the role of Cu^{2+} ion in gold dissolution.

2.2.3 Gold precipitation experiments

After the dissolution of gold in 10 ml of DMSO with 2.0 mmol of $CuBr_2$ and 1.0 mmol of KBr, 2–40 ml of water was added to the solution at ambient temperature. A dilute sulfuric acid aqueous solution, pH = 1, was also used to investigate the effect of pH on the precipitation. After approximately 24 h, the deposited substances were filtered and dried at 353 K, and their weights were measured. The substances were studied using a FESEM (Hitachi S4200) in both secondary electron (working distance of >15 mm, accelerating voltage between 5 and 20 kV) and backscattered electron (BSE, 20 kV) imaging modes, in conjunction with energy-dispersive spectroscopy (EDS) point analysis to determine the qualitative and quantitative element compositions. In some cases, the substances were completely dissolved by aqua regia, nitric acid and perchloric acid. The gold and copper contents were measured by ICP-AES using a Shimadzu ICPS-8100.

3. Results and discussion

3.1 Electrochemical measurement

Figures 1 (a)-(c) show the steady-state polarization curves of gold in DMSO solutions containing $CuBr_2$ and KBr. The rest (corrosion) potentials were -2 mV for 0.1 M $CuBr_2$, +28 mV for 0.2 M $CuBr_2$ and +12 mV for 0.2 M $CuBr_2$ and 0.2 M KBr. Limiting currents were observed for cathodic polarization in all cases at approximately -0.4 V; the currents were 6 mA/cm^2 for 0.1 M $CuBr_2$, 12 mA/cm^2 for 0.2 M $CuBr_2$ and 12 mA/cm^2 for 0.2 M $CuBr_2$ and 0.1 M KBr. Thus, the limiting current density for the cathodic polarization increased proportionally with the increase in the concentration of copper ion in DMSO. However, no limiting current was clearly observed for anodic polarization in any case.

Figure 2 shows the cyclic voltammograms for 0.01 M $CuBr_2$ and 0.4 M tetra-n-butylammonium tetrafluoroborate in DMSO for the glassy carbon electrode, as well as those for 0.4 M tetra-n-butylammonium tetrafluoroborate in DMSO as background measurements. In the presence of 0.01 M $CuBr_2$, cathodic peaks were observed at -65 and -71 mV at scan rates of 50 and 100 mV/s, respectively (labeled C in Fig. 2). After changing the direction of the scan at -0.675 V, the current returned to zero and then showed anodic peaks at +49 and +53 mV at scan rates of 50 and 100 mV/s (labeled A1 in Fig. 2). Other anodic peaks were observed at approximately +0.31 V and +0.34 V (labeled A2 in Fig. 2), which may correspond to the anodic oxidation of DMSO (Krtil, 1996) because these peaks were also observed in the absence of $CuBr_2$. The current densities for the cathodic peaks (C) and anodic peaks (A1) are of nearly the same magnitude. As previously mentioned, both copper (I) and copper (II) ions are moderately solvated in DMSO. Therefore, these peaks can be considered to correspond to the following reaction:

$$Cu^{2+} + e^{-} \leftrightarrow Cu^{+} \quad (1)$$

From the results shown in Fig. 2, the redox potential of reaction (1) was determined to be -8.6 mV for the DMSO solution containing 0.01 M $CuBr_2$. The disproportionation constant, $K_D = [Cu^{2+}][Cu^{+}]^{-2}$, at 298 K was determined to be 2.0 (Chaudhry, 1994). K_D at 343 K can be obtained by the following calculation:

$$K_{D(343)} = \mathrm{Exp.}\ (298/343*\mathrm{Ln}(2.0)) = 1.83 \quad (2)$$

The Nernst equation for reaction (1) is as follows (Izutsu, 2011):

$$E = E^0 + RT/F*\mathrm{Ln}\ ([Cu^{2+}]/[Cu^{+}]) \quad (3)$$

where E^0 is the standard redox potential, R is the universal gas constant and F is the Faraday constant. Therefore, the redox potentials of reaction (1) for DMSO solutions of 0.1 and 0.2 M $CuBr_2$ can be calculated to be +56 mV and +73 mV, respectively, which are more positive compared with the rest (corrosion) potentials of gold shown in Fig. 1. All of these results suggest that the anodic dissolution of gold in DMSO containing $CuBr_2$ occurs at relatively negative potentials and is paired with cathodic reaction (1).

3.2 Leaching gold by DMSO with $CuBr_2$ and bromide salt

Gold was dissolved in 0.1–0.2 M $CuBr_2$ and 0–0.2 M KBr at 333–348 K. Figure 3 shows the changes in the amount of dissolved gold (mmol/L) with time at 348 K. When only 0.1-0.2 M $CuBr_2$ was used, the initial gold dissolution rate was 27 or 37 mg cm^{-2} h^{-1}, respectively, which is relatively large compared with the rates obtained in previous investigations (Lin, 2010; Geoffroy, 2005). The gold dissolution reached equilibrium in 2.0 or 3.5 h at a stoichiometric ratio of dissolved gold to copper of approximately 1:3. When KBr was added to the $CuBr_2$ in DMSO, the dissolution of gold was enhanced. In particular, for the same concentration of $CuBr_2$, the maximum amount of dissolved gold increased by a factor that corresponded approximately to the stoichiometric ratio of the sum of $CuBr_2$ and KBr to KBr.

These results suggest that the dissolution of gold in DMSO containing $CuBr_2$ and KBr occurs via the following anodic electrochemical half-reactions:

$Au(s) + 4Br^- \leftrightarrow AuBr_4^- + 3e^-$ (4)

$Au(s) + 2Br^- \leftrightarrow AuBr_2^- + e^-$ (5)

Thus, the following overall reactions can be obtained:

$Au(s) + 3Cu^{2+} + 4Br^- \leftrightarrow AuBr_4^- + 3Cu^+$ (6)

$Au(s) + Cu^{2+} + 2Br^- \leftrightarrow AuBr_2^- + Cu^+$ (7)

When only $CuBr_2$ is used in DMSO, reaction (6) is dominant, which is supported by the stoichiometric ratio of the maximum amount of dissolved gold to copper, 1:3. In contrast, when KBr is added, reaction (7) also takes place, which results in enhanced gold dissolution with the same amount of Cu^{2+}.

The effect of temperature on the dissolution of gold in DMSO containing $CuBr_2$ and KBr was also investigated at 333–348 K, the results of which are shown in Fig. 4. The dissolution rate of gold clearly increased with increasing temperature. However, the maximum concentration of dissolved gold, 120 mmol/L, did not change significantly with increasing temperature.

Gold dissolution was also conducted in DMSO containing CuBr in argon-purged flasks because no dissolution of gold was observed (Supplementary data). This result indicates that the presence of Cu^{2+} is necessary for gold to dissolve in a DMSO solution containing bromide ion, which supports the notion that the anodic dissolution of gold should be paired with a cathodic reaction (1).

3.3 Precipitation of gold by the addition of water

The precipitation of dissolved gold was performed by the addition of water. When water was added and mixed into the DMSO solution with dissolved gold, precipitation was instantly observed and processed gradually. SEM images of the precipitated substances are shown in Figure 5.

The precipitated substances consisted of clustered primary particles with diameters of 50–200 nm that became smaller when more water was added. Table 1 summarizes the results of the gold precipitation experiments, including the weight of the dissolved gold, the amount of water added to the solution, the total weight of precipitated substances, the amount of precipitated gold, the gold recovery rate and the amount of precipitated copper.

The gold recovery rate was only 52% when 2 ml of water was added, likely because not all of the dissolved gold was precipitated with this ratio of water to DMSO. When more than 5 ml of water was added, the gold recovery rate exceeded 81%. The recovery rate was the largest, 87%, when 5 ml of water was added, and no further increases were observed when more water was added. As shown in Fig. 5, the size of the precipitated particles decreased with increasing amounts of added water. Some of the precipitated gold might have been too fine to be trapped with the filter paper, which could be why the recovery rate did not increase. A small amount of copper was also precipitated with the gold. Figure 6 shows the Eh-pH diagram for copper in the 0.2 M Br^- aqueous solution, which contains a region of stability for dissolved copper, with both $CuBr^+$ and

Cu^{2+} demonstrating stability domains below a pH value of 4. A dilute sulfuric acid aqueous solution, pH = 1, was used for the precipitation of gold. The precipitated substances were analyzed by EDS point analysis, the results of which are shown in Fig. 6. The results indicate that only gold was detected and no copper was observed. These results suggest that the precipitation of copper can be avoided by using water of lower pH and that only gold can be selectively precipitated.

Because WEEE contains a variety of base and precious metals, other metals besides gold may dissolve under certain circumstances and interfere with the efficiency of extraction in leaching processes. Copper is one such metal that has a large share of the weight in secondary sources, e.g., printed circuit boards (Tuncuk, 2012). However, it is expected that the precipitation of copper can be avoided by controlling the pH of water for the selective precipitation of gold in our proposed system.

4. Conclusion

It can be concluded that DMSO solutions of copper bromide ($CuBr_2$) and bromide salts are promising nonaqueous solutions for gold leaching and recovery. The high redox potentials of gold are significantly lowered in DMSO in the presence of bromide ion. Therefore, copper (II) ions in DMSO can act as an oxidizing agent for gold in the presence of bromide ions. The initial gold dissolution rate up to 37 mg cm^{-2} h^{-1} in the DMSO solutions with $CuBr_2$ at 333–348 K was achieved, which is relatively large compared with those obtained with other leaching methods. The dissolution rate can be further improved by increasing the concentration of dissolved $CuBr_2$ and KBr as well as the temperature. Thus, a gold leaching process that is easy to perform and that uses smaller amounts of toxic substances can be developed. The recovery rate of gold up to 87% was obtained by precipitation with water. A small amount of copper was precipitated with gold, which can be avoided by using water of lower pH. This process will enable the leaching solution to be used repeatedly because the water can be easily separated by distillation. Although bromine has been recognized as a potential solvent for gold leaching that offers a number of advantages over cyanide, high bromine consumption has been a bottleneck for its widespread use (Syde, 2013). The repeated use of the leaching solution can overcome this issue. In addition, chemical sludge production can be avoided in this system.

Therefore, a circulating reaction for dissolving and recovering gold with DMSO/water containing $CuBr_2$ and KBr offers a number of advantages, including eco-friendliness, ease of operation, low cost and the minimization of chemical sludge production. Our proposed system is particularly attractive for enhancing the recovery of gold from secondary sources in small- and medium-sized enterprises.

In this paper, leaching and recovering of gold with DMSO/water containing $CuBr_2$ and KBr were proposed and investigated for the first time. Further work should be conducted to investigate the applicability of this system to other precious and rare earth metals.

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Table

Table 1 Summary of gold precipitation experiments

Sample No.	1	2	3	4	5
Dissolved gold (mg)	201	206	199	199	197
Amount of water added (ml)	2	5	10	20	40
Total amount of precipitation (mg)	247	353	303	303	288
Precipitated gold (mg)	105	180	162	168	161
Recovery rate of gold (%)	52	87	81	85	82
Precipitated copper (mg)	37.7	33.2	20.8	16.6	13.8

Figure Captions

Fig. 1 Steady-state polarization curves of gold in DMSO solutions containing $CuBr_2$ and KBr (a) 0.1 M $CuBr_2$, (b) 0.2 M $CuBr_2$, (c) 0.2 M $CuBr_2$ and 0.2 M KBr

Fig. 2 Cyclic voltammograms for 0.01 M $CuBr_2$ and 0.4 M tetra-n-butylammonium tetrafluoroborate and 0.4 M tetra-n-butylammonium tetrafluoroborate in DMSO for a glassy carbon electrode

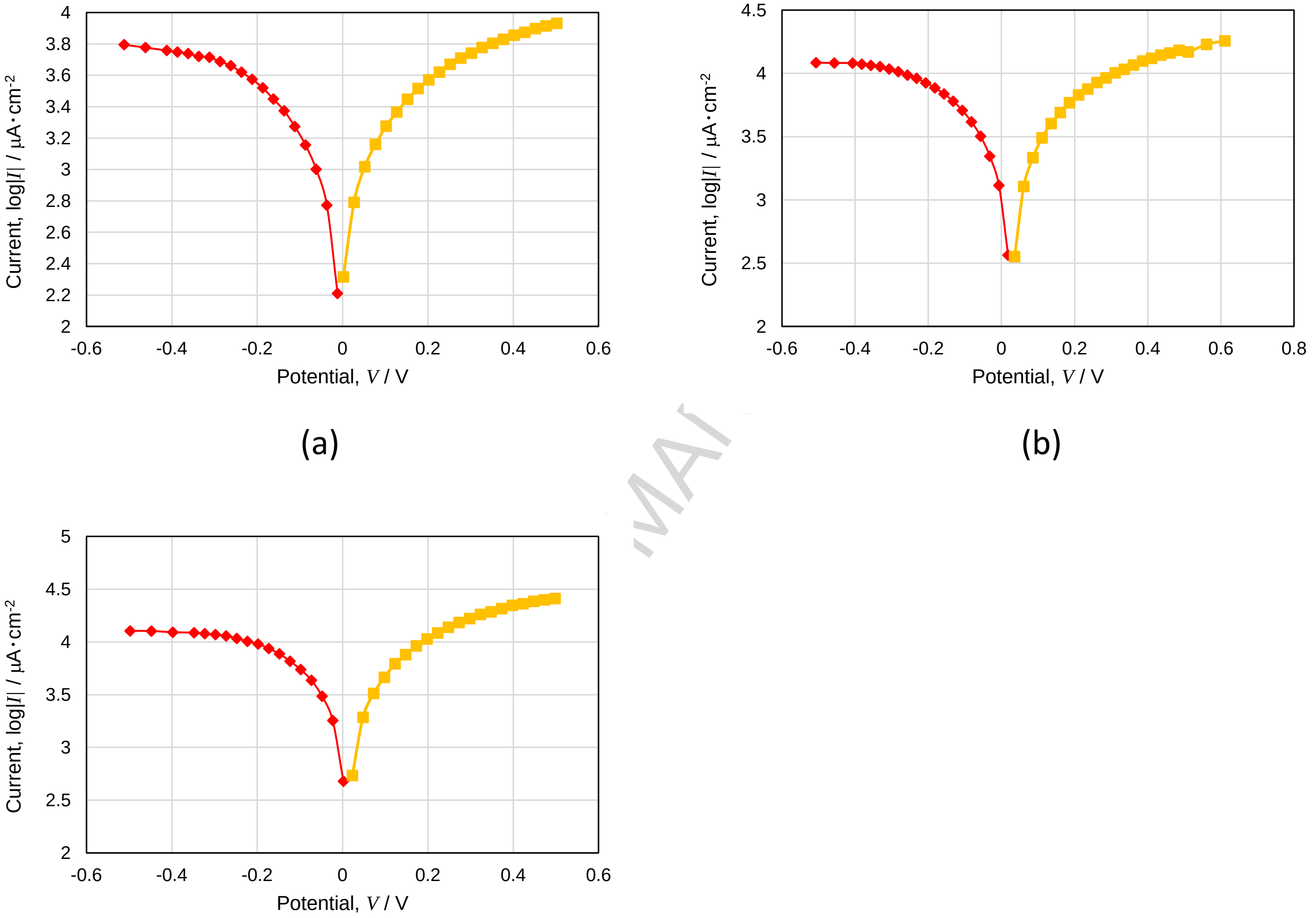
Fig. 3 Changes in amount of dissolved gold with time in DMSO with $CuBr_2$ and KBr at 348 K. The values in brackets in the legend indicate the concentrations of $CuBr_2$ and KBr in DMSO (mmol/L)

Fig. 4 Changes in amount of dissolved gold with time at 333–348 K

Fig. 5 SEM images of the substances precipitated by water

Fig. 6 Eh-pH diagram for copper in 0.2 M Br^- aqueous solution

Fig. 7 EDS point analysis of the precipitated substances by sulfuric acid aqueous solution, pH = 1. The result obtained at Point 4 is shown as an example.

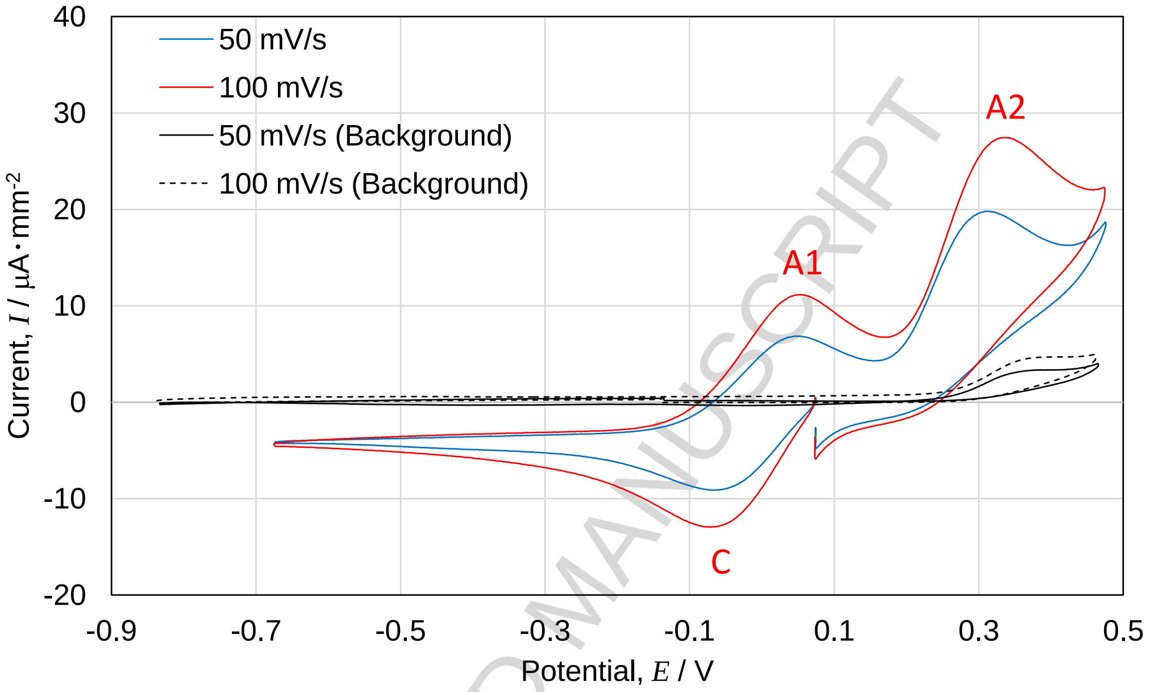


(a)

(b)

(c)

Yoshimura_Fig. 1



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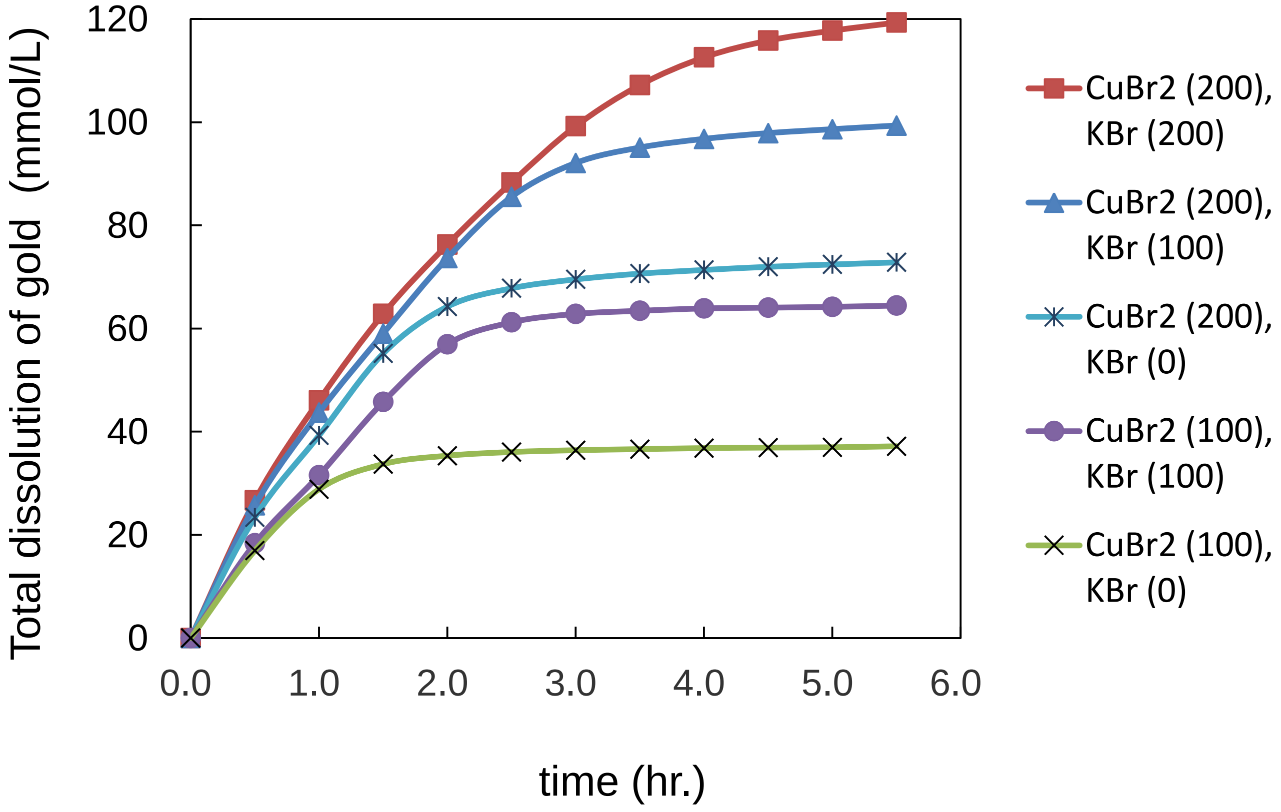
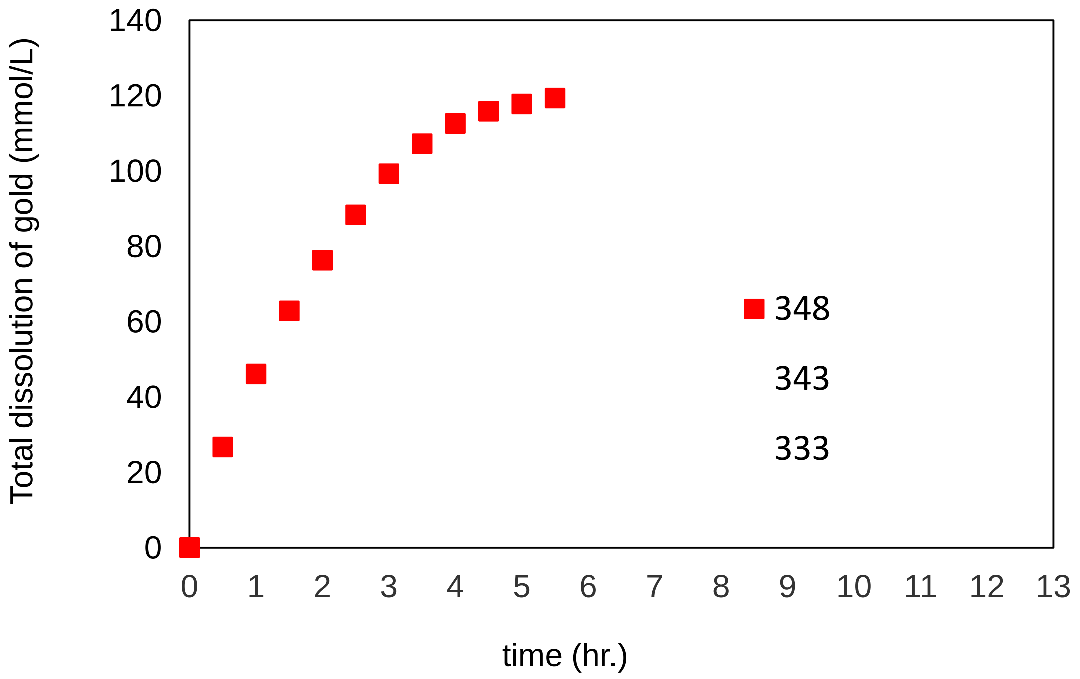
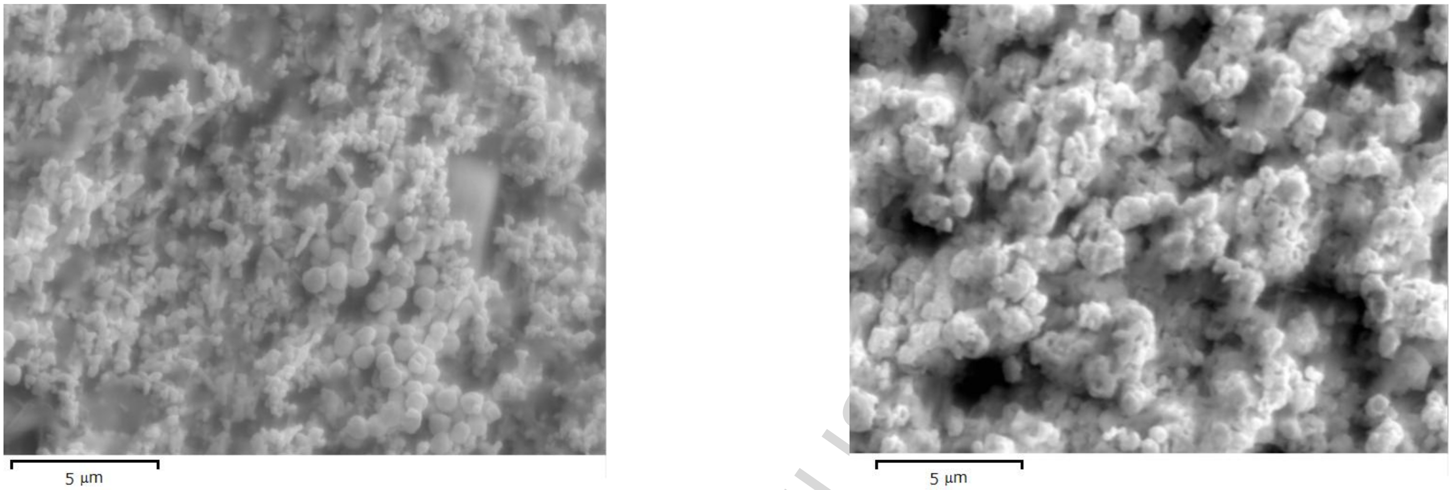


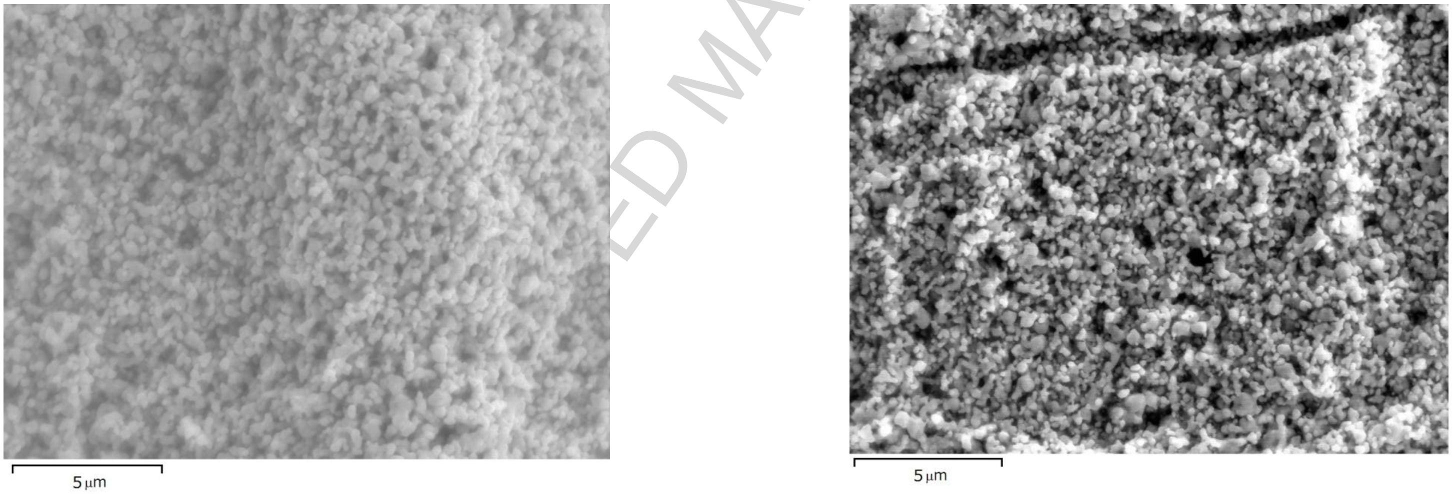
Yoshimura_Fig. 3



Yoshimura_Fig. 4

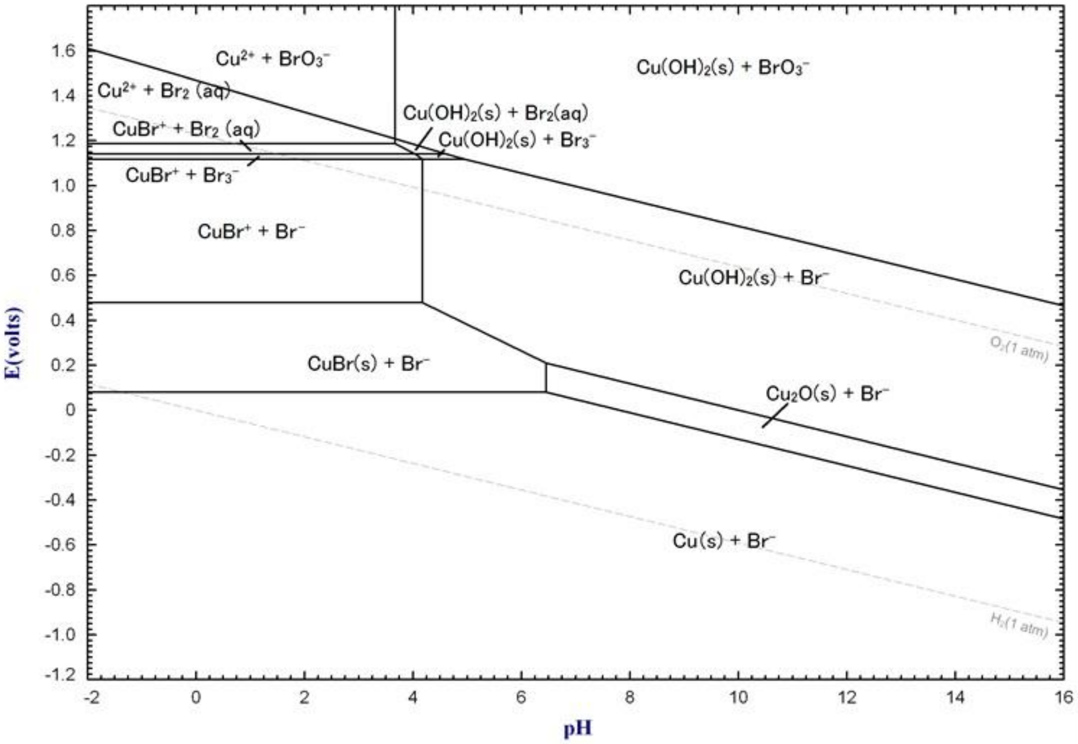


(a) Water: 2 ml (b) Water: 5 ml

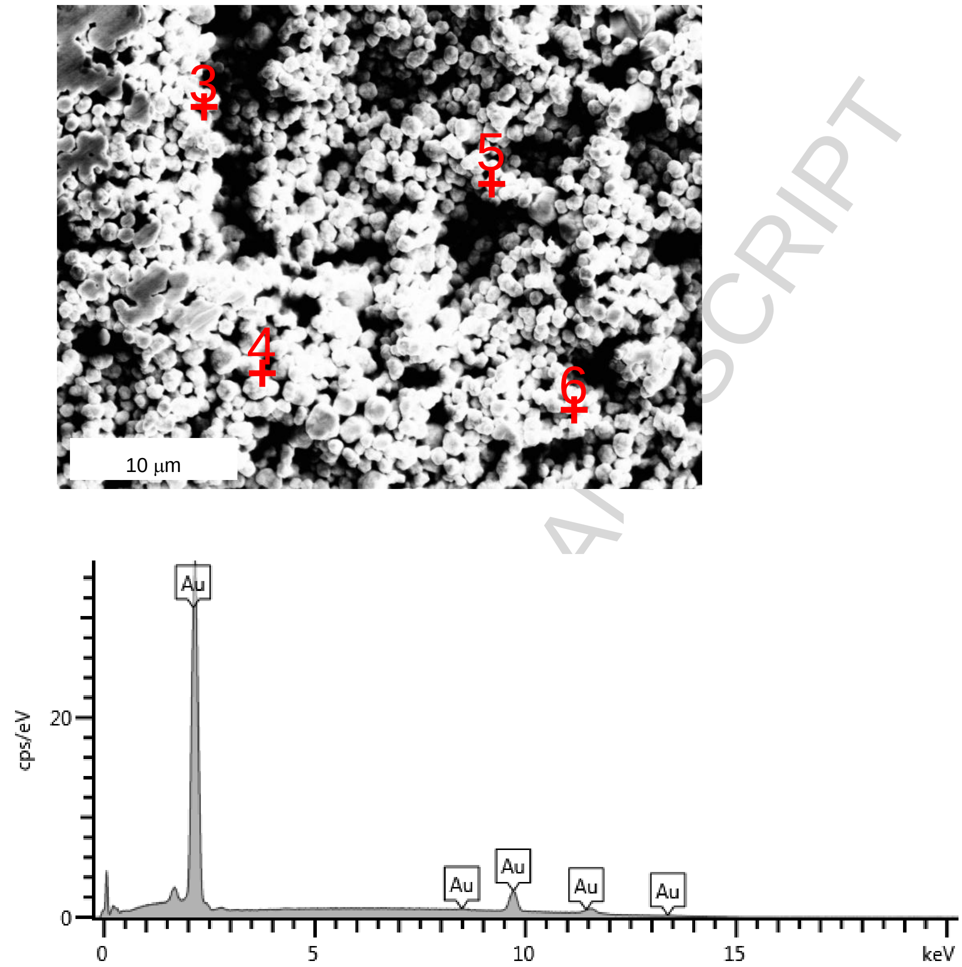


(c) Water: 20 ml (d) Water: 40 ml

Yoshimura_Fig. 5



Yoshimura_Fig. 6



Yoshimura_Fig. 7

Graphical abstract

Highlights

- A novel process for leaching and recovering gold is presented.
- The leaching of gold by DMSO solutions of $CuBr_2$ at a high rate can be achieved.
- The recovery of gold can be easily achieved by precipitation with water.
- Only gold can be selectively precipitated by using water of lower pH.
- A circulating system for gold leaching and recovery can be developed.