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Gold Recovery by pH-Switching Process via Cloud Point Extraction

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ABSTRACT

Poly(oxyethylene)-9-nonylphenyl ether (PONPE-9) was employed for cloud point extraction of gold(III) ion and its phase separation behavior under different experimental conditions was evaluated. The effect on extraction efficiency, including equilibration time, ratio of PONPE-9 to gold, settling temperature, and pH of the solution, was demonstrated. The results indicate that PONPE-9 can be utilized for the extraction of gold in a manner akin to previous work using PONPE-7.5. The optimum experimental results obtained were applied to design a process scheme, pH switching via CPE, to overcome the drawback of the difficulty of separating gold from a gold-entrapped surfactant. The utility

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of the proposed scheme was demonstrated through the application of recovering gold(III) ion from a multimetal solution. Recoveries of greater than 63% of gold were obtained. Even if this is not so high a percentage recovery, it indicates the feasibility of the process scheme. On the other hand, it proved to be an even simpler and easy method for recovering gold from the leaching solution of printed substrates without spending harmful organic solvent.

Key Words: Gold; Cloud point; Extraction; Non ionic surfactant.

INTRODUCTION

Aqueous solutions of polyoxyethylene nonionic surfactants were heated above a certain temperature, the temperature at which clouding occurs, called the cloud point (CP). Above the cloud point, the solution separates into two phases: one, the surfactant-rich phase, which is very small in volume; and the other, the water-rich phase, in which the surfactant concentration is approximately equal to its critical micelle concentration (CMC).[1] Any analytes that can interact with and bind the micellar entity will become concentrated in the surfactant-rich phase upon heating the solution over the CP. This phenomenon, applied for separating some analytes to the surfactant-rich phase, is known as cloud point extraction (CPE).[2] Almost all nonionic surfactants used as mediated surfactants in the CPE studies were isooctyl phenoxy polyethoxy ethanol (TRITON X series) and polyoxyethylene-4-nonyl phenyl ether (PONPE).

This general approach, originally introduced by Watanabe et al,[3] has been utilized for the extraction of nickel in soil and zinc in tap water using PONPE-7.5 and an appropriate chelating agent, namely 1-(2-pyridylazo)-2-naphthol (PAN). The function of the chelating agent is to form an insoluble or sparingly water soluble complex with the metal ion and introduce some selectivity into the process. The scope of the CPE has been extended in the design of the preconcentration of some organic compounds and some metal ions as a step prior to the concentration measurement by HPLC, FIA, and AA. Ahel and Giger[4] studied the preconcentration of alkylphenols and alkylphenol mono- and di-ethoxylates in environmental samples using Triton X-100 as the mediated nonionic surfactant. Garcia Pinto et al[5,6] preconcentrated polycyclic aromatic hydrocarbons and organophosphorus pesticides in the surfactant-rich phase of TRITON X-114 before the measurement by HPLC. Sirimanne et al[7] determined a small amount of polycyclic aromatic hydrocarbons and polychlorinated dibenzo-p-dioxins in human serum after the preconcentration by CPE using

Triton X-100. Furthermore, Akita et al[8,9] also studied the extraction of phenol and its derivatives by the CPE method using PONPE-7.5.

Many works applied CPE to preconcentrate trace amounts of metal ion into the surfactant-rich phase to increase the precision of the instrumental analysis. Garcia Pinto et al[10] employed a complexant system of chelating agent, 1-(2-pyridylazo)-2-naphthol (PAN), and Triton X-114 to entrap chromium ion in the sample and the concentration was subsequently measured. Fernandez Laespada et al[11] determined a small amount of uranium using the complexant system of PAN and Triton X-114. Moreover, the determination of nickel and zinc after preconcentration by CPE using PAN and Triton X-114 was reported by Oliveros et al.[12] It is clear that PAN is not a specific complexant for metal ions. Therefore, an appropriate masking reagent must be employed for practical determination of the target metal ion.[13] Recently, other complexants have been reported to support the determination of some metal ions. Silva et al[14] used the complex form of erbium (III)-2-(3,5-dichloro-2-pyridylazo)-5-dimethylaminophenol to analyze a trace amount of erbium by CPE methodology. Vaidya and Porter[15] determined a small amount of cadmium in water using a chromogenic crown ether in a mixed micellar solution. Mesquita de Silva et al[16] developed the method of determination of silver and gold ions in geological samples by flame atomic absorption spectrometry after the CPE using Triton X-114 mixed with ammonium o,o-diethyldithiophosphate. The different aspects of surfactant-enhanced, nonclassical, metal-extraction schemes, together with the interaction with the micellar entity, were presented by Stalikas.[17] Moreover, the useful information including the basic features, experimental protocols, and selected recent applications of CPE approach were reviewed by Quina and Hinze.[18]

Despite many successful applications of CPE, relatively little work has been devoted to investigate the extraction process scheme for extracting target analytes. Akita et al[19] proposed a scheme of gold ion recovery from printed substrate by CPE using PONPE-7.5 without a complexant. However, it seems to have difficulty in the separation of gold from PONPE-7.5 to obtain the purified metallic gold, and an organic solvent, like chloroform, which was used for stripping gold from PONPE-7.5, was a disadvantage from the viewpoint of harmful inhalation by humans.

Although most works for gold recovery purpose have utilized PONPE-7.5, it was suspected that this type of surfactant with another chain length could be used for the same purpose with a high efficiency. Thus, PONPE-9 was selected because the cloud point of the solution is about 53 ± 0.5°C and the temperature for settling the solution is not too high. The aim of this work was to study the effect of several variables on the CPE efficiency for extracting gold(III) ion from hydrochloric acid using PONPE-9 as the extraction mediator. The variables included the PONPE-9 to gold ion ratio, equilibration time and temperature, and pH of the solution.

EXPERIMENTAL

Materials

Poly(oxyethylene)-9-nonyl phenyl ether ($HO(CH_2CH_2O)_nC_6H_4C_9H_{19}$, PONPE-9) provided by Rhodia (Thailand), Co., Ltd. was used as received. Reagent grade gold(III) chloride solution, $ZnCl_2$, $NiCl_2$, and $CuCl_2 \cdot H_2O$ were purchased from Fluka (Thailand) Co., Ltd. A metal solution was prepared by dissolving an appropriate amount of $HAuCl_4 \cdot 4H_2O$ in dilute hydrochloric acid solution. The solution pH was adjusted with a hydrochloric solution and sodium hydroxide solution. All chemicals used were of reagent grade.

Cloud Point Determination

The cloud point for the different solutions was determined by observing the temperature required for the onset of turbidity upon heating a 4.0-mL aqueous (1.0% w/v) solution of the surfactant in a small test tube that had been placed under a controlled temperature. The temperature was raised in small increments until the clear solution began to cloud. The results reported are the averages of duplicate measurements.

Cloud Point Extraction from Gold Solution

An aqueous solution containing PONPE-9 in the range of 1 to 2.5% (w/v) and gold(III) ion in the range of $(6.3 \text{ to } 25.3) \times 10^{-5}$ M was well mixed at room temperature. Solution pH was adjusted by HCl and NaOH solutions. The mixed solution was then placed in a constant temperature water bath and allowed to separate into two phases at a prescribed temperature for a certain time. The volumes of both the surfactant-rich phase and water-rich phase were read on the volumetric cylinder. The metal content in the aqueous phase was determined by atomic absorption spectrophotometer (Varian Spectra 300/400). The absorption wavelengths for gold(III) ion, copper(II) ion, nickel(II) ion, and zinc(II) ion were 242.8, 324.8, 232.0, and 213.9 nm, respectively. Air–acetylene flame was used for all investigated metals.

The metal concentration of the surfactant-rich phase was calculated on the basis of mass balance.

The extraction efficiency was calculated by the following equation:

$$\%E = 100[M_S/(M_S + M_W)] = 100[V_S C_S/(V_S C_S + V_W C_W)] \tag{1}$$

where C_s equals the concentration of gold(III) ion in surfactant-rich phase; C_w equals the concentration of gold(III) ion in water-rich phase; M_s equals the mass of gold(III) ion in surfactant-rich phase; M_w equals the mass of gold(III) ion in water-rich phase; V_s equals the volume of surfactant-rich phase; and V_w equals the volume of water-rich phase.

It should be noted that the greater the amount of gold in the surfactant-rich phase, the higher the extraction efficiency that could be obtained.

Cloud Point Extraction from Multimetal Solution

The separation of gold from a multimetal solution was also studied at various solution pH values. A mixed metal ion solution of copper(II), nickel(II), zinc(II), as well as gold(III) ions was prepared with a composition as shown in Table 1. This solution is representative of printed substrate and dilute liquor. CPE was conducted under optimum conditions. The metal contents in both the water-rich phase and surfactant-rich phase were determined by atomic absorption spectrophotometer. The percent extraction of each metal thus can be calculated according to Eq. (1).

Table 1. Metal contents of printed substrate and diluted liquor used as multimetal solution feed.

	Gold(III) ion	Copper(II) ion	Nickel(II) ion	Zinc(II) ion
Aqueous feed for this work (ppm)	5	100.9	8.25	0.08
Substrate (mg/g-solid)[a]	5.9	119.8	9.8	0.09
Aqueous feed for gold recovery process (ppm)	59	1183.6	98.8	0.9

[a] According to Akita et al.[17]

Gold Recovery Process by CPE in 1-L Column

The CPE of gold was also performed in a 1-L settle column. The inner glass column, with a diameter of 12.0 cm, has a valve for draining solution at the bottom and the outer acrylic column of 20.0-cm diameter was circulated with water of constant temperature.

First, the mixed metal ion feed solution (see Table 1) was passed to extraction column number 1. The CPE at the optimum conditions (settling temperature = 65°C, solution pH = 3, ratio of PONPE-9 to gold = 1500) was conducted to trap gold ion into the surfactant-rich phase. When the phase separation equilibrium was reached, the surfactant-rich phase was transferred to extraction column number 2 and adjusted to pH 7. The CPE was conducted at the minimum efficiency conditions obtained from the experimental results (settling temperature = 65°C, solution pH = 7). It should be noted that the CPE conducted in extraction column number 2 was for removing gold from the surfactant. Thus, the chosen operating condition was the condition that yielded the lowest percentage extraction of gold in the surfactant-rich phase. The water-rich phase was then separated to the precipitation bath where metallic gold was precipitated using ascorbic acid as a reducing agent (mole ratio of gold:ascorbic acid = 1:10). The gold-free surfactant was reloaded in the process.

Scanning Electron Microscopic Study

A scanning electron microscope (SEM) mounted with an electron dispersion x-ray analyzer (EDX) was used for investigating the fineness of the surface and analyzing the percent purity of precipitated gold. The gold powder was carefully mounted on an SEM stub using double-sided tape and then examined under microscope model JSM-6400, operated at 20 kV.

RESULTS AND DISCUSSION

Effects of Equilibration Time

It should be mentioned that a lot of research on CPE allowed a long period of time, such as overnight, to make sure that the separation reached the equilibrium separation between phases. Therefore, the time required for allowing the equilibrium separation between phases was one of the important parameters that suggested the feasibility of the process scheme. The time of equilibration was optimized by finding the shortest time period required for complete separation of the phases. In this series of experiments, the concentration of PONPE-9 was kept constant at 1% (w/v) and the gold concentration was 1 or 2 ppm. The pH of the solution was in the range of 2.52 to 2.89. The temperature was controlled at 56, 60, or 65°C. The results of these experiments are presented in Fig. 1. In Fig. 1, C_1 is the concentration of gold ion in the water-rich phase and C_0 is the initial concentration of gold ion in the solution. From Fig. 1, the ratio of C_1/C_0 decreases rapidly with time and reaches an equilibrium. At the settling temperatures of 56, 60, and 65°C, the results show the effect of temperature on the separation rate between phases. The higher the settling temperature, the shorter period of time required to reach separation equilibrium between phases. This is attributed to the dehydration rate that depends on the settling temperature. It is known that heat supplied to the surfactant solution causes dehydration from the surfactant moiety, consequently, with surfactant aggregation and separation of surfactant from the aqueous phase. Therefore, the dehydration rate depends on the amount of energy gained from supplied heat. Then, the aggregation rate of the surfactant into the surfactant-rich phase increases with increasing temperature. All curves suggest a required time of 6 hours for the complete separation between phases.

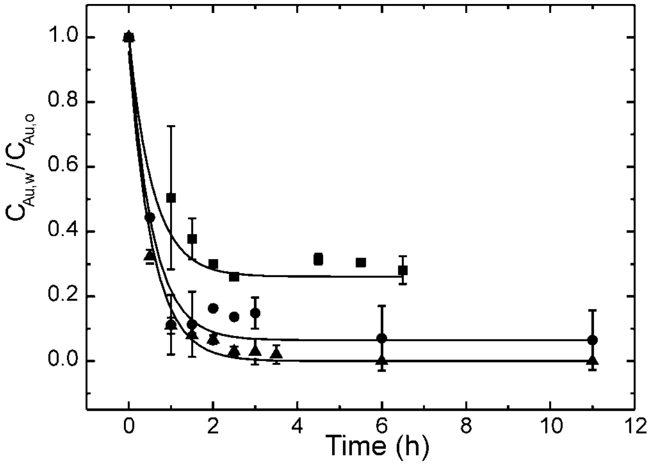

Figure 1. Effect of equilibration time on $C_{Au,w}/C_{Au,o}$ at T = 56°C (■), T = 60°C (●), T = 65°C (▲). $C_{Au,o}$ is the initial concentration of gold ion in the mixed solution, $C_{Au,w}$ is the concentration of gold ion in the water-rich phase at any time, [PONPE-9] = 1% (w/v), [Au^{3+}] = 1, 2 ppm, pH = 2.52–2.89.

Effect of PONPE-9 to Gold Ion Ratio

A series of experiments was performed in which the gold concentration was varied over the range of 1 to 18 ppm. The PONPE-9 concentration was 1 or 2.5% (w/v). The temperature was kept constant at 60°C. The solution pH was in the range of 2.52 to 2.89. The results are shown in Fig. 2. It is shown that the percent extraction of gold increases with increasing the ratio of PONPE-9 to gold from 400 to 1500. At a PONPE-9 to gold ratio higher than 1500, the percentage extraction showed insignificant changes. The ratio of the surfactant PONPE-9 to gold that provides the highest efficiency (98.78%) with a small amount of surfactant is about 1500.

Effect of Settling Temperature

Experiments were carried out from 56 to 80°C with a PONPE-9 concentration of 2.5% (w/v). The gold concentration was 5 or 10 ppm. The pH of the solution was adjusted to 1.97 and 2.98. The results of the dependence of the percentage extraction of gold on the settling temperature are depicted in Fig. 3. The percentage extraction of gold initially increases, levels off, and

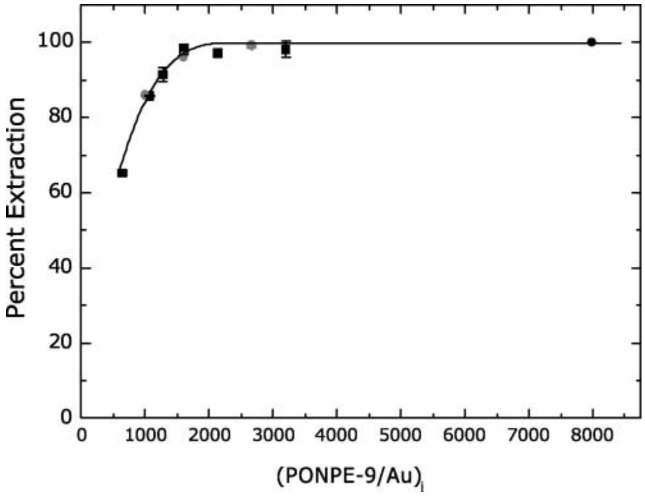

Figure 2. Effect of ratio of PONPE-9 and Au^{3+} on percentage extraction. [PONPE-9] = 1% (w/v) (■), [PONPE-9] = 2.5% (w/v) (●), $[Au^{3+}] = 1-18$ ppm, pH = 2.52 to 2.89, T = 60°C, t = 6 h.

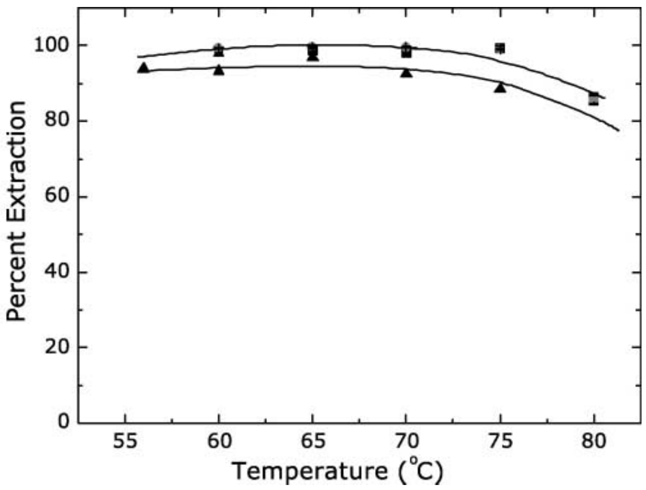

Figure 3. Effect of temperature on percentage extraction. [PONPE-9/Au^{3+}] = 1599 at pH = 2.98 (■), [PONPE-9/Au^{3+}] = 1599 at pH = 1.97 (●), [PONPE-9/Au^{3+}] = 799 at pH = 2.98 (▲), t = 6 h.

then decreases as the settling temperature increased. A similar pattern of temperature effect has been reported for the extraction of gold with HCl and PONPE-7.5 and the extraction of 1-hexanol with Igepal CA-620.[2,20] This behavior is ascribed to poor phase separation upon too small or too large a temperature difference between the CP and settling temperature.[19] From Fig. 3, it is seen that a settling temperature in the range of 65 to 70°C provides a high percentage extraction of gold. The range of the settling temperature could substantiate the conclusion published elsewhere that the greatest effect of the equilibration temperature on the extent of extraction occurs when the equilibration temperature is less than 15 to 20°C greater than the cloud point temperature.[2] Therefore, for the highest percentage (99.0%) with lower energy consumption, the settling temperature of 65°C is appropriate for the gold recovery scheme.

Effect of Solution pH

An interesting result was obtained in the study of the effect of solution pH. In this series of experiments, the solution pH was varied over the range of 1 to 12. The PONPE-9 and gold concentrations were kept constant at 2.5%

(w/v) and 5 ppm, respectively (the ratio of PONPE-9 to gold is 1599). The experiments were carried out at the settling temperatures of 56, 65, and 80°C. The obtained percentage extraction of gold is shown as a function of solution pH in Fig. 4. It was found that all experiments conducted in either acidic and basic solutions provided a high percentage of extraction with a maximum at a pH of 3; ca. 98 to 99% of gold. This is in sharp contrast to gold extraction conducted at pH 7, where only 21 to 25 percentage extraction can be achieved. This result suggests a pH influence on the gold complex moiety involving the interaction between gold-complex ion and PONPE-9. At pH 7, the solution contains an equal amount of hydrogen ion and hydroxide ion, providing a neutral form of solute, while an ionic form of the gold-complex ion exists in the acidic and basic solutions. The results at pH 7 suggest that the neutral form of the gold-complex ion normally does not interact with and bind the micellar aggregate as strongly as does its ionic form. Therefore, a lesser amount of gold ion is extracted in the surfactant-rich phase and the percentage of extracted gold decreases at pH 7.

Therefore, the solution pH affects the surfactant and the gold-complex ion structures. It is known that the gold(III) ion exists in hydrochloric acid as tetrachloro gold(III) ion at a low pH,[21] while gold(III) hydroxide forms at a high pH. Both forms of the gold complex with an ionic moiety can bind with

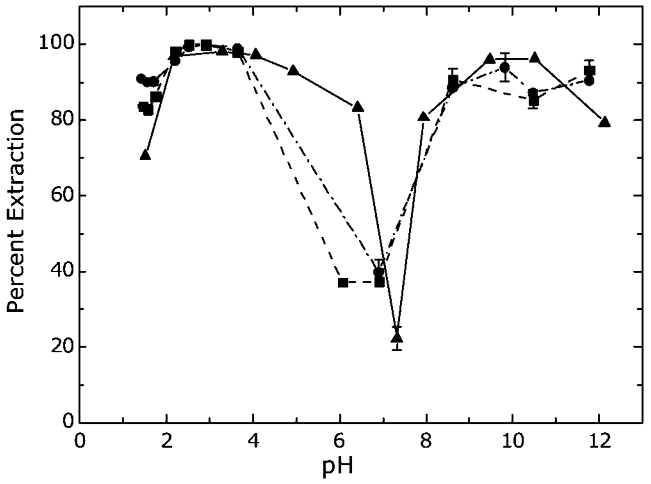

Figure 4. Effect of pH on percentage extraction at T = 56°C (■), T = 60°C (●), T = 80°C (▲). [PONPE-9] = 2.5% (w/v), $[Au^{3+}]$ = 5 ppm, $[PONPE\text{-}9/Au^{3+}]$ = 1599, t = 6 h.

PONPE-9 in micelle aggregation. It is controversial at pH 7, where both the gold-complex ion and surfactant exist in the neutral moiety. Thus, the interaction of PONPE nonionic surfactant and the metal ion was driven by the ionic interaction between the complex ion and the high-electron affinity atoms of PONPE.

Though the extraction of some target analytes to the surfactant-rich phase is not difficult, the removal of entrapped analytes from the mediated surfactant is not easy. From this experimental result, the CPE at pH 7 can be used as the method of separation of the gold-complex ion from PONPE-9.

From this work, the appropriate conditions for the CPE of gold(III) ion are as follows:

1. Equilibration time for separation between phases is 6 hours.
2. The ratio of PONPE-9 to gold ion is 1500:1.
3. The settling temperature is 65°C.
4. The maximum and minimum efficiency for recovering gold ion are at the solution pH values of 3 and 7, respectively.

A mixed metal solution of copper(II), nickel(II), zinc(II), as well as gold(III) ions (see Table 1) was used. The extraction was performed at 65°C

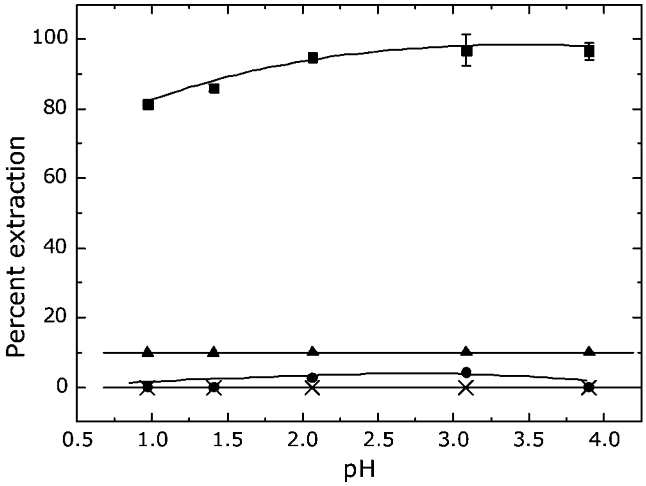

Figure 5. Effect of pH on percentage extraction of metal ions. Au^{3+} (■), Ni^{2+} (▲), Cu^{2+} (●), Zn^{2+} (×), [PONPE-9] = 2.5% (w/v), $[Au^{3+}]_o = 5$ ppm, $[Cu^{2+}]_o = 100.9$ ppm, $[Ni^{2+}]_o = 8.25$ ppm, $[Zn^{2+}]_o = 0.08$ ppm, pH = 1–3.8, T = 65°C, t = 6 h.

and the ratio of PONPE-9 to gold ion was 1500. Figure 5 shows the effect of the pH on the percentage extraction of each metal. It can be seen that the extraction of other metals is depressed and complete extraction of gold (98.8%) is obtained in the same manner as for the single metal system at the same pH region (see Fig. 4). The percentage extraction of gold (80 to 95%) at lower pH (pH 1 to 2) is less than that at pH 2 to 3.9 (98.8%). Little change in the percentage extraction of the undesirable metals was observed for the investigated pH range. Thus, a solution pH of 3 was chosen to be the operating condition for the extraction of gold from multimetal solution. The obtained percentage extractions of gold, copper, nickel, and zinc were 98.8, 4.3, 10.0, and 0.0%, respectively.

Gold Recovery Process by CPE

As a result of the effect of variation of the solution pH, a large amount of gold was extracted into the surfactant-rich phase, while the amount extracted was very small at pH 7. The result at pH 7 suggests suitable conditions for

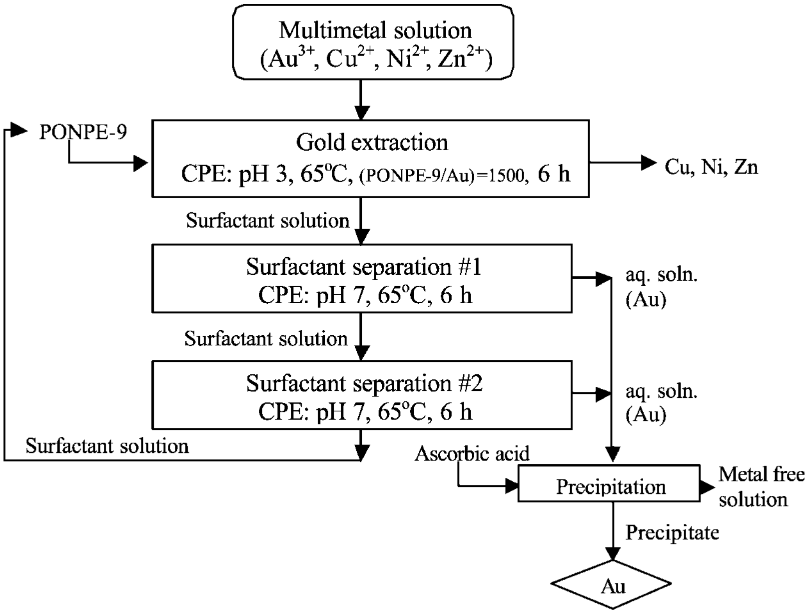

Figure 6. Process scheme of gold(III) ion recovery using CPE with PONPE-9.

separating gold from gold-entrapped surfactant. The process scheme for recovering gold from multimetal solution was designed as shown in Fig. 6. The process mainly consists of three steps: (1) extraction of gold ion from multimetal solution into the surfactant-rich phase, (2) separation of gold from gold-entrapped surfactant, and (3) precipitation of gold metal.

Figure 7 shows the gold extraction in the extraction column number 1. The mixed solution appears as the clear blue color of copper ion, as shown in Figure 7a. It is noteworthy that the mixed metal solution contains much larger amounts of copper and nickel than of gold. Figure 7b shows the turbid phenomena at pH 3 after the increase in temperature to the CP (65°C). Figure 7c and 7d illustrate the two phases at equilibrium separation after 6 hours. Here, the gold ion is extracted into the surfactant-rich phase (bottom phase), which shows the yellow color of gold, while other ions (Cu^{2+}, Ni^{2+}, and Zn^{2+}) exist in the water-rich phase (upper phase), which shows a blue color.

After the surfactant-rich phase was transferred into separation column number 2, water was added and the solution pH was adjusted to pH 7 (Fig. 8a). The CPE was then applied at a temperature of 65°C for 6 hours. It should be noted that the gold(III) ion surrounded with hydroxide ion shows a purple color. Figure 8b illustrates the separation between phases after the CPE at pH 7. It shows a purple color of the water-rich phase enriched with gold ion, while the gold-free surfactant settles in the surfactant-rich phase with a white color. Percentage recovery of gold in the water-rich phase was 63.3.

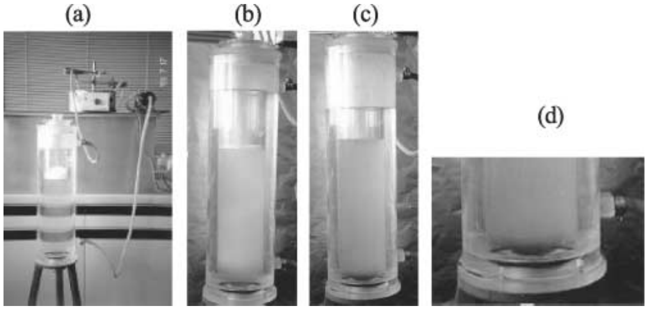

Figure 7. Photograph of the experiment on gold extraction from multimetal solution: (a) the initial multimetal solution in settling column number 1 before heating, (b) turbid phenomena after increasing the temperature to the CP, (c) the two phases after equilibrium separation, and (d) close-up picture showing the interface between surfactant-rich and water-rich phases.

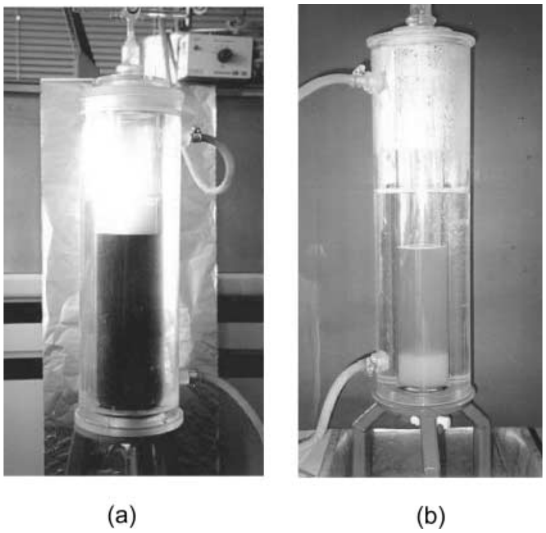

Figure 8. Phase separation after CPE in the column number 2.

The gold solution was next transferred to the precipitation bath and precipitated by ascorbic acid as the reducing agent.[19] The experimental results for the percentage recovery of gold are summarized in Table 2. The 63.3 percentage recovery of metallic gold was obtained with 98.01% purity, as determined by SEM mounted with EDX. Figure 9 shows the scanning electron micrograph of the surface of the high fineness gold powder precipitated.

Table 2. Percentage extraction of gold from the multimetal solution.

Column number	Operation	Condition	Cycle number	Percentage of gold
1	Gold extraction	pH = 3 T = 65°C Time = 6 h [PONPE-9/Au^{3+}] = 1500	1	98.8
2	Gold separation from surfactant-rich phase	pH = 7 T = 65°C Time = 6 h	2	63.3

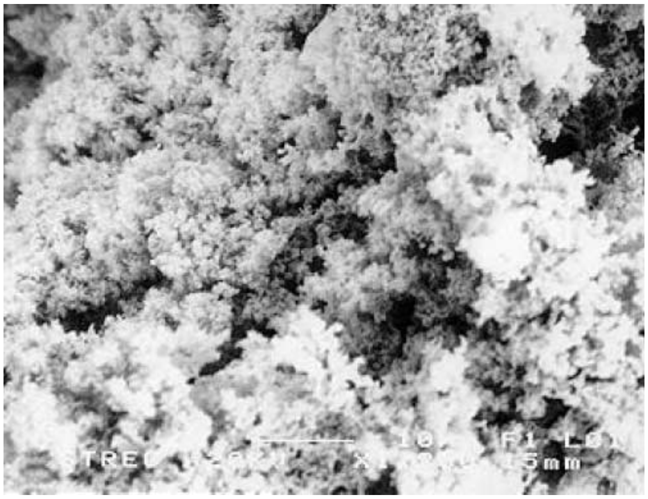

Figure 9. Scanning electron micrograph of precipitated gold metal (× 1000).

CONCLUSION

PONPE-9 provides good extraction efficiency for recovering gold ion from multimetal solution. The proposed pH-switching process scheme has two batches of CPE at different solution pH values. The first batch is for extracting gold ion from other metal ions at pH 3. The second is conducted at pH 7 for separating gold ion from gold-entrapped surfactant. Metallic gold is finally precipitated. Advantages cited for the use of the pH switching method include (1) the extraction can be carried out with no use of organic solvents for metal stripping and (2) after the extraction of gold from other metal ions by applying CPE at pH 3, it is much easier (compared with organic solvent extraction) to separate gold by subsequently applying CPE at pH 7. Moreover, the separation efficiency could be improved by introducing a multistage process, which deserves further study. Therefore, this is a choice for gold ion recovery from discarded electronic parts under environmental conservation concepts.

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