Cloud-Point Extraction of Gold(lll) with Nonionic Surfactant—Fundamental Studies and Application to Gold Recovery from Printed Substrate

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ABSTRACT

An aqueous solution of a nonionic surfactant, polyoxyethylene nonyl phenyl ether, having an average of 7.5 oxyethylene units, provides an aqueous two-phase system that is surfactant-rich and surfactant-lean at ambient temperature. The system found use as a separation field. Au(III) was effectively partitioned into the small surfactant-rich phase from hydrochloric acid solutions; thus such operational factors as the concentrations of the surfactant, HCl, and NaCl were investigated. Partitioning of other heavy metals was depressed, and selective extraction of Au(III) could be attained. Such a cloud-point extraction technique has been applied to the recovery of gold from an aqua regia leaching solution of a printed substrate, and an overall process scheme, including stripping and reduction steps, was proposed. By introducing scrubbing stages using a dilute HCl solution, satisfactory recovery of gold with a high yield and purity from undesirable metals could be obtained.

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INTRODUCTION

Increasing demand for gold in recent industrial products such as electric devices and catalysts makes it crucial to recover gold from the inevitably increasing waste products from the viewpoint of resource conservation. Since conventional selective dissolution-conditioning and precipitation processes for recovering precious metals are labor-intensive, more flexible hydrometallurgical ones such as solvent extraction (1) and ion exchange (2) have attracted special attention.

Cloud-point extraction is a relatively novel separation method based on an aqueous two-phase system with polyethylene-glycol-type nonionic surfactants. Aqueous solutions of nonionic surfactants become opaque at a definite temperature referred to as the cloud point (CP), and are subject to phase separation by settling at a temperature above the CP. Such a phenomenon results from the dehydration of ethylene oxide (EO) units in the surfactants at elevated temperatures with a subsequent decrease in aqueous solubility. One of the phases formed, the coacervate phase, is rich in the surfactant concentration and its volume is usually much smaller than the other bulk aqueous phase. CP extraction is founded on the partitioning of a solute which has an affinity for the surfactant between the water-based two phases.

CP extraction has been applied for separating some organic compounds from aqueous media (3–10); however, its application to metal separation is limited (11). In a previous paper (12) we reported the CP extraction of Au(III) from HCl media with polyoxyethylene nonyl phenyl ether having an average EO unit of 10 (PONPE10). It was found that phase separation from aqueous surfactant solutions could be attained by heating; Au(III) was successfully recovered into the small surfactant-rich phase. Increasing the HCl concentration caused a reduction in Au(III) extraction as a result of a rise in the CP. However, extraction was improved at a high surfactant concentration, at a high settling temperature (ST), or by adding NaCl. We studied the phase-separation characteristics of aqueous PONPE solutions from both the equilibrium and kinetics viewpoints (13).

The driving force for phase separation of nonionic surfactant solutions depends on the solution temperature. In our previous work (12) the ST was kept mainly at 70°C. If phase separation can be attained at a lower temperature, such as the ambient temperature, it might be an advantage from the viewpoint of process economics. In such a situation the surfactant solutions would be in a state of phase separation from the beginning of the operation, and hence the methodology of CP extraction would be similar to that of conventional solvent extraction, including the availability of existing extraction equipment.

Nonionic surfactants containing a shorter EO unit in the molecule are known to have lower solubility in aqueous media due to less hydrophilicity

with a consequent decrease in the CP. In the present study we have fundamentally investigated the CP extraction of Au(III) from HCl media by the use of PONPE7.5 to develop an aqueous two-phase system which operates at ambient temperature. We also made a study on the applicability of CP extraction to the recovery of gold from a printed substrate as an industrial waste.

EXPERIMENTAL

Reagents

Polyoxyethylene nonyl phenyl ethers (PONPE) with average numbers of ethylene oxide (EO) unit of 5, 7.5, 10, and 15 were purchased from Tokyo Kasei Kogyo and used as nonionic surfactants without further purification. The general formula of PONPEn is $HO(CH_2CH_2O)_nC_6H_4C_9H_{19}$, where n is the EO number. A stock metal solution was prepared by dissolving an appropriate amount of each metal chloride of $HAuCl_4 \cdot 4H_2O$, $PdCl_2$, $H_2PtCl_6 \cdot 6H_2O$, $ZnCl_2$, $CuCl_2 \cdot 2H_2O$, and $FeCl_3 \cdot 6H_2O$ in a dilute HCl solution. All chemicals used were of reagent grade.

Procedure

CP extraction of metals from synthetic metal solutions was carried out as follows. A desired feed solution was prepared by mixing a prescribed amount of PONPE and/or NaCl with appropriate volumes of standardized HCl and a stock metal solution, then adding deionized water to bring the total volume of 20 cm^3. The initial concentrations of the surfactant and each metal in the aqueous feed were fixed at 2.5 wt% and 5×10^{-4} M, respectively, unless otherwise noted.

An aqueous feed (20 cm^3) was mixed vigorously in a 25-cm^3 graduated glass tube and heated slowly in a thermostated water bath; the temperature at which the solution became turbid was recorded as the CP. After the CP had been observed, the solution was kept standing at a prescribed settling temperature (ST) overnight in a thermostated water bath or an incubator in order to achieve phase separation. The equilibrium volumes of both the surfactant-rich and bulk aqueous phases were measured. The metal concentration in the aqueous phase was determined by inductively coupled plasma spectroscopy (ICP), and that in the surfactant-rich phase was calculated on the basis of the mass balance. In the case of an aqueous phase with a very low metal concentration, however, the concentration in the surfactant-rich phase was directly measured after solubilizing it in water by adding a small amount of PONPE15 to minimize the experimental error.

The experimental procedures and conditions for downstream processing (scrubbing, stripping, precipitation) of the CP extraction will be presented in the following section.

Metal Leaching from Printed Substrate

A printed substrate, kindly supplied by Hokkai Can Co., consisted of a plastic substrate overprinted with gold and base metals as a metal strip. Leaching was carried out by immersing small pieces of the substrate (ca. 1.0 cm × 1.0 cm) in aqua regia in a glass beaker overnight at room temperature. The ratio of the substrate to the solution was kept at 125 g/dm^3. The elemental metal contents of the substrate were determined from a mass balance.

RESULTS AND DISCUSSION

Phase Separation of Nonionic Surfactant Solutions

Figure 1 shows the effect of HCl concentration on the CP of aqueous PONPE solutions containing a small amount of Au(III) (5 × 10^{-4} M). The CP monotonously rises with an increase in the HCl concentration, so that a higher ST is required to attain phase separation. This surfactant salting-in with the acid might be attributable to the interaction between oxonium ion and oxygen atom in the surfactant EO unit, which makes the surfactant micelles more hydrophilic. In contrast to HCl, the addition of NaCl causes a lowering of the CP, viz., salting-out of the surfactants, through the competition for hydrated water between the electrolyte and the surfactant (14).

The PONPE7.5 system gives a much lower CP than PONPE10 because the former surfactant has less hydrophilicity due to its shorter EO unit. For
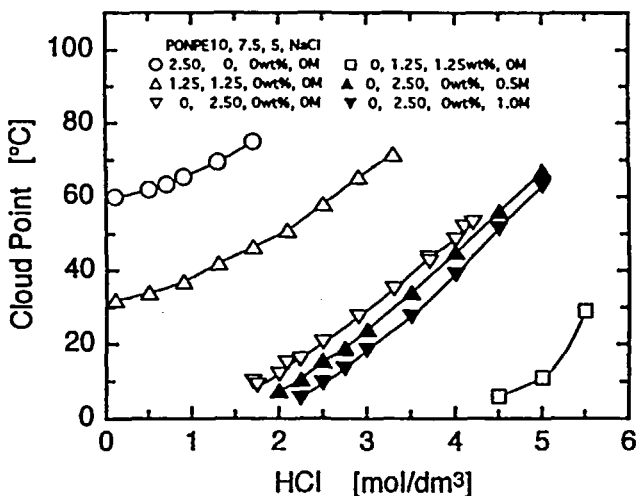

FIG. 1   Effect of HCl concentration on cloud point of aqueous PONPE solutions.
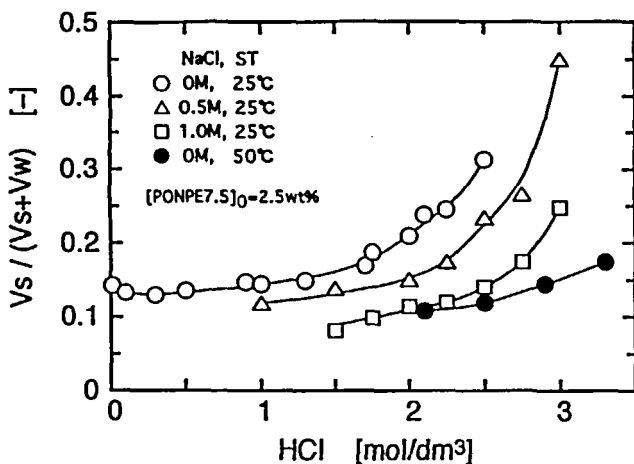
FIG. 2   Effect of HCl concentration on volume ratio of surfactant-rich phase.

the mixed surfactant system of PONPE10 and PONPE7.5, the CP lies between those for each single system, and the mixed system of PONPE7.5 and PONPE5 gives an even lower CP, even at higher HCl concentrations. Such a CP lowering leads to the possibility of CP extraction at either high HCl concentration or low ST.

Figure 2 represents the effect of HCl concentration on the equilibrium volume ratio of the surfactant-rich phase, $V_s/(V_s + V_w)$, where V denotes the volume and the subscripts s and w are the surfactant-rich and -lean aqueous phases, respectively. In the present system the surfactant-rich phase was formed as a lower phase. With increasing HCl concentration the volume ratio increases and the CP rises. However, a further increase caused poor phase separation because the temperature difference between the CP and ST became too small. On the other hand, the addition of NaCl not only leads to a decrease in the volume ratio but also to possible phase separation from solutions with higher HCl concentrations.

A rise in the ST enables CP extraction to be applied for solutions with higher acidity. However, clear phase separation was not observed at 50°C for solutions with HCl less than 2 M. This implies that too large a temperature difference between the CP and ST (over 30°C) is also not appropriate for phase separation from PONPE solutions (13). Moreover, the volume ratio was observed to increase proportionately with an increase in the feed PONPE7.5 concentration, although the data are not shown here.

Figure 2 shows that the volume of the surfactant-rich phase is much smaller than that of the aqueous bulk phase, thus, in CP extraction, the concentration

of a target solute can be expected to become higher compared with conventional solvent extraction. It should also be noted that the amount of surfactant remaining in the bulk aqueous phase after phase separation is close to the surfactant's critical micelle concentration (CMC) (13). Thus, the loss of surfactant in CP extraction is so small as to be negligible.

Cloud-Point Extraction of Gold(III)

Fundamental Parameters of CP Extraction

The CP extraction of Au(III) from HCl media was evaluated in terms of the percent extraction, E, and the distribution ratio, D, defined as follows:

$$E = 100[M_s/(M_s + M_w)] = 100[V_sC_s/(V_sC_s + V_wC_w)] \qquad (1)$$

$$D = C_s/C_w \qquad (2)$$

where M and C denote the amount and concentration of metal, respectively. Here we have adopted the percent extraction based on the amount of metal in the two phases instead of the concentration, since the volume ratio varies with the experimental conditions.

Figure 3 shows typical results for the CP extraction of Au(III) with PONPE7.5 at 25°C, together with that for PONPE10 at 70°C without NaCl (dashed line). Note that a high extraction of Au(III) can be attained without the addition of any complexing agents into the system. Because Au(III) exists
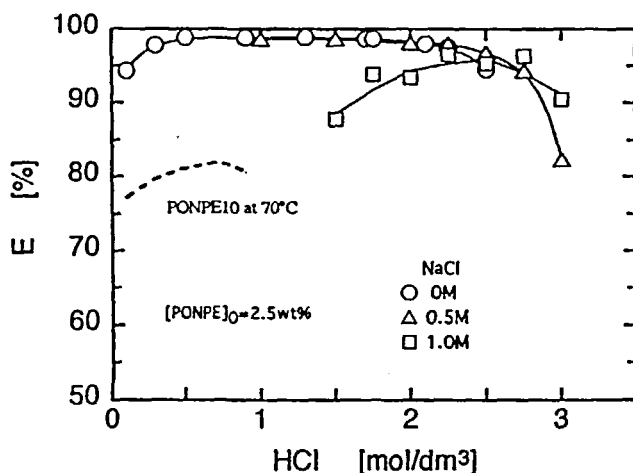

FIG. 3   Effect of HCl concentration on percent extraction of Au(III) at 25°C.
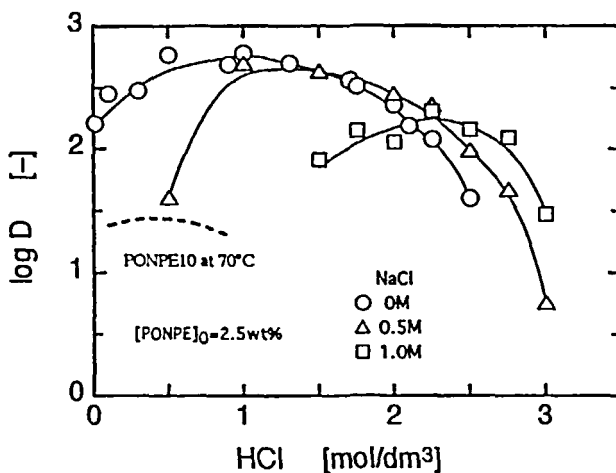
FIG. 4   Effect of HCl concentration on distribution ratio of Au(III) at 25°C.

in HCl solutions in the form of $AuCl_4^-$, extraction might take place through coordination of the surfactant EO unit to chloroauric acid, $HAuCl_4$, akin to solvent extraction of Au(III) with solvating extractants.

The PONPE7.5 system not only provides a higher extraction efficiency than the PONPE10 system but also has a wider HCl concentration range favorable for extraction. Such behavior may be attributable to an exothermic process in the complex formation between Au(III) and the EO units in the surfactant molecule. Furthermore, on adding NaCl, the CP is lowered and the HCl concentration favorable for extraction shifts to a higher region. However, it should be noted that in some cases the addition of salt results in poor phase separation. In particular, a solution with 1.0 M NaCl resulted in ambiguous phase separation, with the surfactant-rich phase being formed as an upper phase in the region of high HCl concentration.

Figure 4 shows the effect of HCl concentration on the distribution ratio of Au(III). The D values are so large that a high concentration of Au(III) in the surfactant-rich phase can be expected. The PONPE7.5 system gives a much higher distribution ratio than the PONPE10 system (dashed line); however, the ratio tends to decrease in regions of both lower and higher HCl concentrations. This behavior might be ascribed to bad phase separation due to too small or too large a temperature difference between the CP and ST as described in the previous section.

Figures 5 and 6 show the effect of PONPE7.5 concentration on the percent extraction and the distribution ratio of Au(III), respectively. An increase in
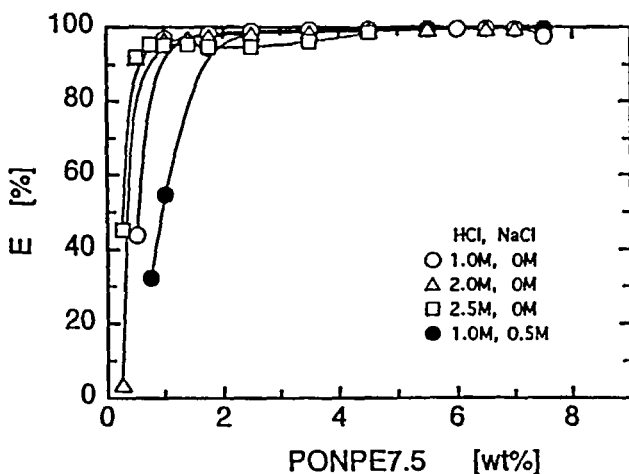
FIG. 5   Effect of surfactant concentration on percent extraction of Au(III) at 25°C.
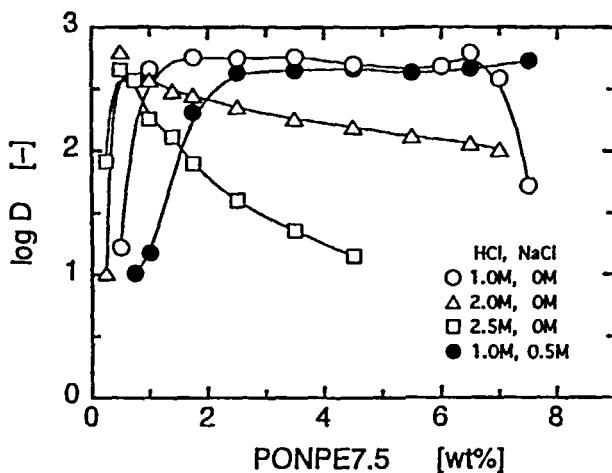

FIG. 6   Effect of surfactant concentration on distribution ratio of Au(III) at 25°C.
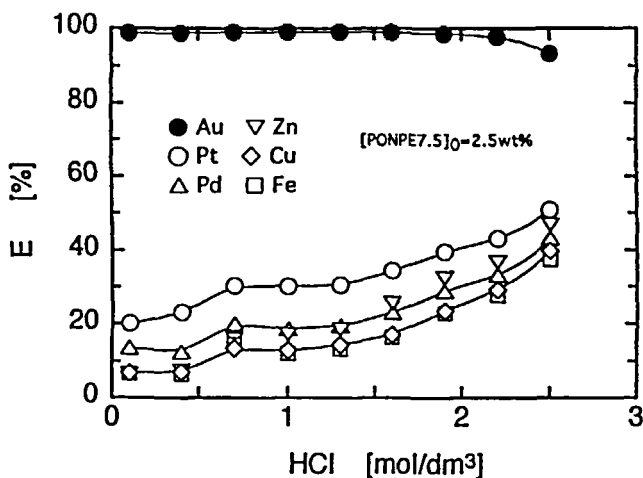
FIG. 7   Effect of HCl concentration on percent extraction of heavy metals.

the surfactant concentration provides a dramatic increase in the extraction efficiency, and almost complete extraction can be attained with concentrations larger than 1.5 wt%. In the case of systems without salt, at any HCl concentrations the D values increase with an increase in the surfactant concentration up to 0.5 wt%, corresponding to 9.1 mM and ca. 18 times the initial Au(III) concentration. For surfactant concentrations of more than 1 wt%, however, the distribution ratio tends to decrease, and the dependency is more significant at higher acidities. Such a decline can be interpreted in terms of an increase in the volume of the surfactant-rich phase.

Optimization of the system parameters allows a distribution ratio over 500; thus, CP extraction provides an efficient method for the recovery of Au(III) from HCl media. However, it should be noticed that an excess of surfactant might lower the solute concentration in the surfactant-rich phase (see Fig. 6), unlike solvent extraction where the volume ratio of the two phases is independent of the extractant concentration.

Separation of Au(III) from Multimetals Solution

Metal separation via CP extraction using PONPE7.5 was studied at 25°C for an aqueous multimetals solution of platinum(IV), palladium(II), zinc(II), copper(II), and iron(III) as well as gold(III). Figure 7 shows the effect of HCl concentration on the percent extraction of each metal. Although the extraction of the other metals is depressed, complete extraction of Au(III) is almost
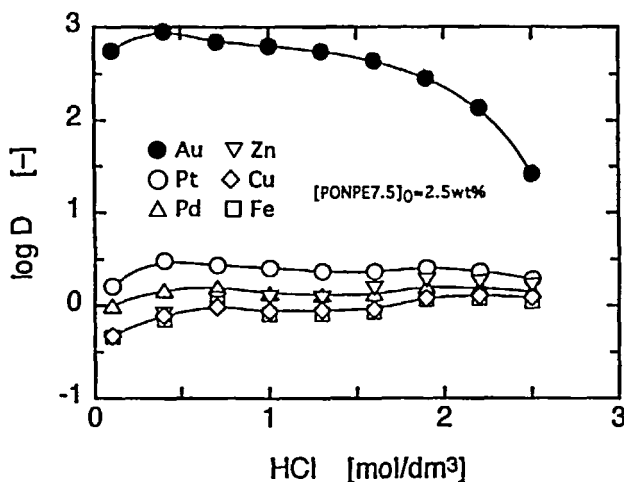
FIG. 8   Effect of HCl concentration on distribution ratio of heavy metals.

attained in the same manner as that for the single metal system (see Fig. 3), and selective separation of Au(III) can be obtained. With increasing HCl concentration, however, the E values of the undesirable metals tend to increase along with an increase in the volume of the surfactant-rich phase; thus, there is a decrease in selectivity.

The distribution ratio of each metal is plotted in Fig. 8 as a function of the HCl concentration. In contrast to the high distribution ratio of Au(III), the log D values of the other metals are around zero, independent of the HCl concentration. It should be noted that the nonselective metal ions are equally partitioned between the two phases, and hence can not be excluded from the surfactant-rich phase. This leads to a strong dependence of the selectivity on the volume ratio of the two phases. Thus, in CP extraction it is important to retain operating conditions which yield a small volume of the surfactant-rich phase from the viewpoints of both solute concentration and separation.

Table 1 lists the separation factors of Au(III) with some heavy metals, $\alpha_{Au/M}$, from 1.0 M HCl media with and without NaCl, defined as

$$\alpha_{Au/M} = D_{Au}/D_M \qquad (3)$$

where the subscript M represents the undesirable metals in the present situation. The following sequence of extraction selectivity was found: Fe(III) < Cu(II) < Zn(II) < Pd(II) < Pt(IV) ≪ Au(III). The addition of NaCl to the system had a negative effect on Au(III) selectivity. Thus, CP extraction with

TABLE 1

Separation Factors of Au(III) with Respect to Some Metals

NaCl (M)	$\alpha_{Au/Pt}$	$\alpha_{Au/Pd}$	$\alpha_{Au/Zn}$	$\alpha_{Au/Cu}$	$\alpha_{Au/Fe}$
0	248	465	518	727	771
0.5	192	344	337	437	503

PONPE7.5 provides both efficient separation and a high concentration of Au(III) from acidic media.

Downstream Processing of Cloud-Point Extraction

Scrubbing of Surfactant-Rich Phase

To improve the separation of Au(III) from other heavy metals, CP extraction was repeated by adding a metal-free aqueous solution to the surfactant-rich phase from the previous step. Such a treatment is thought to correspond to scrubbing in conventional solvent extraction.

Table 2 gives the percent recovery of the each metal in the surfactant-rich phase before and after scrubbing, where a scrubbing solution was added into the surfactant-rich phase (ca. 2.8 cm^3) from the first CP extraction with 1.0 M HCl to a total volume of 20 cm^3. Extraction was then repeated in the same manner. Although very high recovery of Au(III) was attained in the first CP extraction, appreciable amounts of the undesirable metals were also entrapped into the surfactant-rich phase. However, these metals were dramatically decreased by scrubbing with 1.0 M HCl, and therefore an improvement in gold purification could be achieved. This is because only Au(III) is preferentially

TABLE 2

Enhancement of Metal Separation by Scrubbing[a]

Operation	HCl (M)	% Recovery in surfactant-rich phase					
		Au	Pt	Pd	Zn	Cu	Fe
CP extraction	1.0	99.4	24.4	15.7	12.8	10.6	10.9
1st scrubbing	1.0	98.6	6.8	2.3	1.6	0.9	1.1
2nd scrubbing	1.0	97.2	2.2	0.1	0.1	N.D.	0.1
CP extraction	1.0	99.5	24.4	15.7	12.8	10.6	10.9
1st scrubbing	0	97.9	4.8	1.9	1.1	0.8	0.6
2nd scrubbing	0	91.9	0.9	N.D.	N.D.	0.0	N.D.

[a] The results are an average of two determinations. N.D. = metal not detected.

TABLE 3
Recovery of Au(III) from Surfactant-Rich Phase[a]

Operation	Agent	% Au(III) in aqueous solution		
		0.1 M[b]	0.5 M[b]	1.0 M[b]
PONPE extraction	CHCl$_3$ (5 mL)	67.3	34.1	20.8
Metal stripping	0.1 M HCl (5 mL)	31.0	51.3	57.5
Total recovery	—	98.3	85.4	78.3
PONPE extraction	CHCl$_3$ (10 mL)	72.7	43.9	25.9
Metal stripping	0.1 M HCl (5 mL)	27.3	50.2	58.9
Total recovery	—	100	94.1	84.8

[a] The results are an average of two determinations.
[b] HCl concentration for CP extraction.

extracted into the surfactant-rich phase, and the other metals, which have no affinity for the surfactant, are equally partitioned between the two newly formed phases as described earlier. Further enhancement of separation can be easily attained by applying multiple scrubbing. The recovery of Au(III) was as high as 97.2%, even for double scrubbing.

Scrubbing with pure water leaves smaller amounts of the undesirable metals in the surfactant-rich phase than with 1.0 M HCl. Only Pt(IV) remained in a significant amount after double scrubbing. Such an enhancement in separation is ascribed to lower extraction of the undesirable metals at lower HCl concentrations (see Fig. 7). Unfortunately, the washout of Au(III) into the scrubbing solutions tends to increase with a lowering of acidity; in the double scrubbing with water, Au(III) recovery in the surfactant-rich phase was decreased to 91.9%.

Stripping of Au(III) from Surfactant-Rich Phase

In a previous study (15) on the solvent extraction of Au(III) with PONPE as an extractant in chloroform, we found that the extraction efficiency decreases with decreasing HCl concentration. This finding led to the development of a procedure for removing only PONPE7.5 from the surfactant-rich phase into the organic phase, with Au(III) left in the aqueous solution.

Extraction of PONPE from the Au(III)-entrapped surfactant-rich phase (ca. 2.8 cm^3) was studied using CHCl$_3$ (5 or 10 cm^3), and the results are summarized in Table 3 where the percentage of Au(III) recovered in the aqueous solution is reported. The Au(III) concentration in the original surfactant-rich phase was ca. 4×10^{-3} M for all runs, and the mixing and settling times of the two phases were 5 minutes and 3 hours, respectively. Most of the

PONPE7.5 was extracted into the organic phase, while an appreciable amount (67.3%) of Au(III) remained in the aqueous phase, as expected. During surfactant transfer the volumes of the organic and aqueous phases varied from 5.7 to 5.7 cm^3 and from 2.8 to 2 cm^3, respectively. During PONPE extraction the residual Au(III) in the aqueous solution was found to decrease from 67.3 to 20.8% with HCl concentration increasing from 0.1 to 1.0 M. This behavior is responsible for the entrainment of Au(III) with the surfactant into the organic solution. Poor phase separation and metal precipitation were observed during PONPE extraction with HCl solutions lower than 0.1 M.

To strip the Au(III) entrained with the surfactant from the organic phase, 0.1 M HCl (5 cm^3) was again brought into contact with its solution, and the results are also shown in Table 3 as "Metal stripping." When the two solutions are mixed (3 minutes) and settled (overnight), an appreciable amount of Au(III) (from 31.0 to 57.5%) was found to be stripped into the aqueous solution. Thus, high recovery of Au(III) from the surfactant-rich phase can be attained by adding together the aqueous solutions in the first (PONPE extraction) and second (metal stripping) operations. Moreover, increasing the CHCl$_3$ volume from 5 to 10 cm^3 yielded a higher percentage Au(III) in the first step. Thus, complete recovery can be achieved in the case of the CP extraction with 0.1 M HCl.

Reuse of the surfactant extracted in CHCl$_3$ was also examined by evaporating the solvent to leave PONPE7.5. No significant decline in extractability due to this treatment was observed, with negligible loss of the surfactant. This indicates that the small amount of Au(III) entrained with PONPE in the organic solution after surfactant extraction can be returned to the process by recycling the surfactant.

Reduction of Au(III)

To obtain metallic gold, precipitation of Au(III) from dilute HCl solutions was studied by chemical reduction using ascorbic or oxalic acid, and the results are summarized in Table 4. A prescribed amount of the reducing agent was added into the HCl solution containing 2.2×10^{-3} M Au(III). The resulting solution (5 cm^3) was allowed to stand for 18 hours at 70°C in an incubator. The precipitate was filtered out, and the yield and purity of the precipitate was calculated from the difference in the metal concentration in the solution before and after precipitation. In the case of ascorbic acid, almost complete precipitation was achieved, even with 0.004 M agent [less than twice the initial Au(III) concentration], independent of the HCl concentrations studied. In contrast, oxalic acid gives a much lower yield, depending on the HCl concentration; thus, a larger amount of the reagent will be needed to obtain a high yield.

TABLE 4
Precipitation of Au(III) Using Two Reducing Agents

		% Precipitation		
Reducing agent		0.1 M HCl	0.5 M HCl	1.0 M HCl
Ascorbic acid	0.004 M	99.7	100	99.9
	0.01 M	99.9	100	100
	0.02 M	100	99.8	100
Oxalic acid	0.004 M	87.6	7.5	7.4
	0.01 M	98.4	25.8	7.4
	0.02 M	100	48.0	10.3

Direct reduction of Au(III) in the surfactant-rich phase was also done using the same reducing agents. Although the reaction probably proceeded to precipitation based on the color change, it was impossible to separate the precipitate from the solution, even by centrifugation.

Direct electrodeposition of Au(III) from the surfactant-rich phase has been tried. Unfortunately, since the limiting current density for Au(III) reduction is greatly decreased in the presence of PONPE7.5, we could not obtain a satisfactory yield. This low efficiency may be due to a strong interaction between the surfactant and the metal. A possible alternate method for recovering metals from the loaded surfactant-rich phase is by direct burning of the liquid; this strategy might be especially advantageous for a high loading of valuables.

Recovery of Gold from Printed Substrate

On the basis of the experimental results described in the previous sections, we attempted to construct a process for recovering gold from a printed substrate via CP extraction with PONPE7.5. The overall flow scheme is illustrated in Fig. 9.

Leaching of metals from a printed substrate was conducted using aqua regia, and the metal contents of the substrate are listed in Table 5. Completion of leaching was confirmed by the disappearance of the metal strips on the substrate. It is noteworthy that the substrate contains copper and nickel in much larger amounts than gold and the separation of gold from them may be crucial in the process.

After filtering the plastic matrix, PONPE7.5 was added in the leach liquor and then diluted with deionized water to an acidity of ca. 1 M to allow high efficiency in the subsequent CP extraction (see Fig. 7). A part of this solution (20 cm^3) was used as an aqueous feed for the following steps, where the
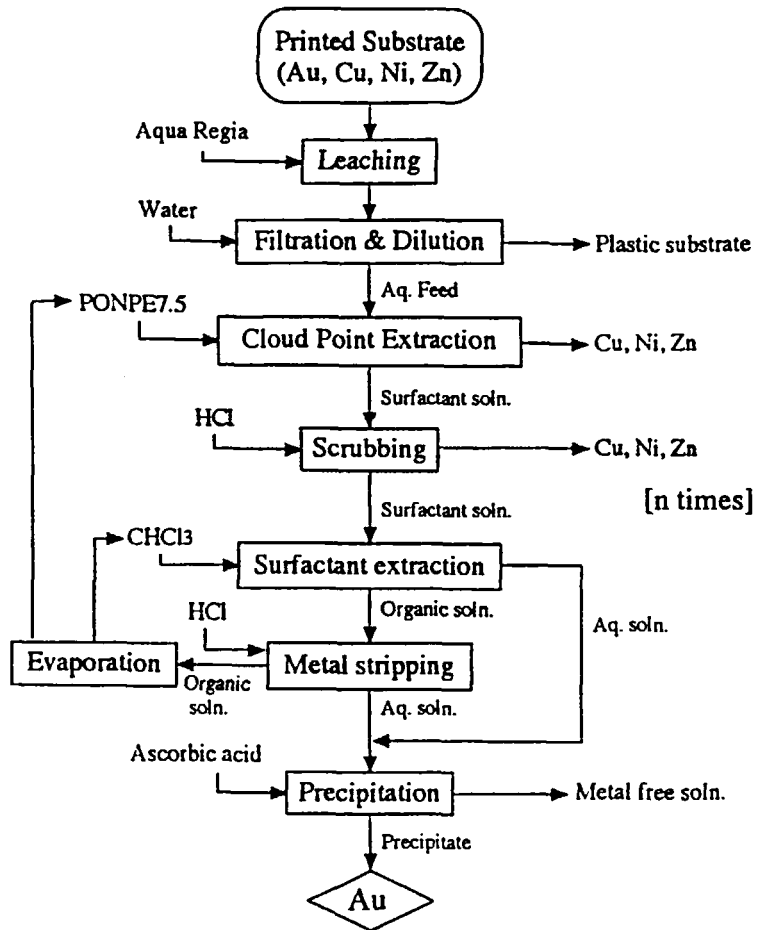
FIG. 9   Process scheme for recovery of gold from printed substrate.

TABLE 5
Metal Contents of Printed Substrate and Diluted Leach Liquor Used as Aqueous Feed

	Au	Cu	Ni	Zn
Content of substrate (mg/g-solid)	5.9	119.8	9.8	0.09
Concentration in aqueous feed (ppm)	93.2	1880.5	154.0	1.48

surfactant concentration was set to be 2.5 wt% and the metal concentrations are also listed in Table 5.

The procedures used here were similar to those described earlier. After conducting the CP extraction at 25°C, the resultant surfactant-rich phase (2.8 cm^3) was scrubbed with 1.0 M HCl (17.2 cm^3) a maximum of three times. Then, PONPE7.5 was extracted by CHCl$_3$ (10 cm^3) from the surfactant-rich phase, whereby the volumes of the organic and aqueous solutions changed into 10.5 and 2.3 cm^3, respectively. The metals entrained with the surfactant were subsequently stripped out with 0.1 M HCl (5 cm^3) from the organic phase. The resultant two aqueous solutions were added together (7.3 cm^3), and metallic gold was obtained by reducing this solution with ascorbic acid (0.01 M).

The experimental results for the percentage recovery of the each metal are summarized in Table 6. Here, the proposed process scheme is examined in terms of a cycle number of the scrubbing (n), where the three recoveries are presented for (1) the surfactant-rich phase after CP extraction and/or scrubbing, (2) the aqueous solution after PONPE extraction and metal stripping, and (3) the precipitate. Even without scrubbing ($n = 0$), the undesirable metals can be depressed to less than 10% in the final precipitate, though the gold purity was as low as 32.0% because of the high feed concentrations of copper and nickel. On repeating the scrubbing, the separation of gold is im-

TABLE 6
Recovery of Gold from Printed Substrate

Operation	n^a	% Recovery (% purity)			
		Au	Cu	Ni	Zn
1. CP extraction	0	98.8	11.4	11.6	7.6
2. SE-stripping		86.1	10.7	10.9	7.6
3. Precipitation		86.1(32.0)	9.0	9.2	7.4
1. Scrubbing	1	98.1	1.2	1.1	N.D.
2. SE-stripping		82.6	1.1	1.1	N.D.
3. Precipitation		82.6(80.8)	0.9	0.9	N.D.
1. Scrubbing	2	97.3	0.1	0.1	N.D.
2. SE-stripping		83.1	0.1	0.1	N.D.
3. Precipitation		83.1(97.3)	0.1	0.1	N.D.
1. Scrubbing	3	96.0	0.0	N.D.	N.D.
2. SE-stripping		78.0	0.0	N.D.	N.D.
3. Precipitation		78.0(99.8)	0.0	N.D.	N.D.

^{a} Cycle number of scrubbing.

proved; purity as high as 99.8% can be attained in the triple operation ($n =$ 3). It should be noted that a relatively high loss of gold is found in the SE-stripping step. However, most of the gold retained with PONPE7.5 can be recycled into the process, as mentioned in the previous section. By taking account of such recycle operation, the total loss of gold is a satisfactory low of 4.0%, even for triple scrubbing.

The gold concentration in the each step in the process is as follows: 93 ppm in the feed, 617 ppm in the surfactant-rich phase after triple scrubbing, and 201 ppm in the stripping solution before precipitation. Although these values are not as high as expected, the concentration would be improved by treating a larger volume of the feed solution with a small amount of the surfactant.

There was little difference between the results for the synthesized solution and for the leaching liquor from the actual sample. Moreover, the process was confirmed to be feasible for feed volumes as large as 1000 cm^3. Thus, the proposed flow scheme seems to be a novel candidate for recovering gold from a printed substrate.

CONCLUSIONS

Cloud-point extraction of Au(III) with PONPE7.5 has been studied from the viewpoints of both fundamentals and applications. The following conclusions were reached.

1. Phase separation of aqueous PONPE7.5 solutions can be obtained at ambient temperature with HCl concentrations below 3.0 M.
2. Au(III) is successfully extracted into the surfactant-rich phase from dilute HCl solutions. The maximum concentration of Au(III) is obtained at around 1.0 M HCl, and it shifts to higher acidities when NaCl is added.
3. Extraction is improved by increasing the surfactant concentration. However, excessive surfactant (over 1.0 wt%) lowered the Au(III) concentration.
4. The selective CP extraction of Au(III) from several heavy metals is possible from HCl solutions up to 2.5 M.
5. Gold recovery from a printed substrate by the use of a process flow chart (see Fig. 9) gave satisfactory recovery of gold in the presence of excessive amounts of undesirable metals. A final purity of 99.8% with the loss as little as 4.0 % could be obtained with three scrubbing stages.

Compared with conventional solvent extraction, CP extraction is superior for concentration but inferior for separation. The present technique might replace conventional separation methods in some situations, including hydrometallurgy and bioseparations.

NOMENCLATURE

C concentration (mol/dm^3)
D distribution ratio defined by Eq. (2) (—)
E percent extraction defined by Eq. (1) (%)
M amount (mol)
V volume (dm^3)
α separation factor defined by Eq. (3) (—)
[]   concentration (mol/dm^3), (wt%)

Subscripts

M   metal
s   surfactant-rich phase
w   aqueous phase
0   initial

ACKNOWLEDGMENTS

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