

## Predispersed Solvent Extraction of Negatively Complexed Copper from Water Using Colloidal Liquid Aphron Containing a Quaternary Ammonium Salt

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**Abstract**—Negatively complexed copper ion by complexing agent like EDTA (Ethylenediaminetetraacetic acid) was removed by predispersed solvent extraction (PDSE) using colloidal liquid aphrons (CLAs) made out of Trioctylmethylammonium chloride (Aliquat 336) diluted with nonpolar kerosene. PDSE was found to have higher mass transfer rate than conventional solvent extraction under experimental conditions without mechanical mixing. The effect of type of water-soluble surfactants, phase volume ratio (PVR), concentration of anionic Sodium Dodecyl Benzene Sulfonate (SDBS) on PDSE was investigated. In addition, the effect of anionic SDBS on back extraction in PDSE was also studied. Under experimental conditions with enough mechanical mixing, the amount of copper transferred to Aliquat 336 core from the pregnant phase was compared in both PDSE by using anionic SDBS and conventional solvent extraction. It is concluded that PDSE using Aliquat 336 CLA can be used for treatment of negatively complexed copper without the influence of surfactant. To optimize CLAs-based process, stability of CLAs containing a quaternary ammonium salt Aliquat 336 diluted with kerosene in the continuous phase was investigated by measuring the volume released to surface. To destabilize CLAs,  $H^+$ ,  $OH^-$  were added. Stability of CLAs was estimated by comparing the half-life obtained. Break-up of destabilization follows pseudo-first-order reaction kinetics at low ionic strength. But, pseudo-first-order model cannot be applied to a region of high ionic strength.

Key words: Colloidal Liquid Aphron, Predispersed Solvent Extraction, Copper, Trioctylmethylammonium Chloride (Aliquat 336), Stability

### INTRODUCTION

In order to minimize the effect on human beings, heavy metals such as copper must be removed from effluent of industrial process. Major effluent sources of heavy metals include electrodeless copper plating process for printed circuit boards, washing effluent of metal-contaminated soils and electrode metal plating process [Spearot and Peck, 1984]. Solvent extraction using various extractants can be used to remove heavy metals selectively, and can be applied to recover heavy metals by back-extraction [Seader and Henley, 1998]. However, conventional solvent extraction has an energy consuming mixer-settler and requires a large volume of extractants for treatment of metals. Predispersed solvent extraction (PDSE) using colloidal liquid aphrons (CLAs) can overcome this shortcoming of conventional solvent extraction. An aphron is defined as a distinct unit of fluid encapsulated by a thin soapy film of another fluid. High concentrations of these aphrons, up to 96% dispersed in 4% of aqueous phase, are termed polyaphrons. As water is the continuous phase, when a polyaphron is added to water it disperses to give CLAs. This soapy film enables polyaphrons containing extractants to be dispersed in the pregnant phase without mechanical mixing. Therefore, conventional mixer-settler stages can be eliminated and energy can be saved by comminuting only one phase instead of both pregnant phases containing substances to remove and organic phases containing extractants. In addition, the enhancement of mass transfer rate can be

achieved due to these micro-sized polyaphrons by increasing interfacial area [Zhang et al., 1996]. The schematic structure of an aphron and the preparation mechanism of polyaphrons are shown in Fig. 1.

Since PDSE using CLAs was proposed by Sebba [Sebba, 1985, 1986, 1987], it has been applied into the removal of hydrophobic organic compounds [Lee et al., 2000], the recovery of organic acid from fermentation process [Kim et al., 2002], oil recovery [Sebba, 1985, 1987], recovery of proteins in bioprocess [Lye and Stuckey, 1996], the removal of some metals [Sebba, 1985, 1987]. The removal of positive non-complexed copper ion employing CLAs was first studied by Save [Save et al., 1994]. They observed that mass transfer rate can be increased and the amount of organic solvent can be reduced by PDSE using polyaphrons containing LIX 622. However, this LIX 622 cannot be used to remove negatively complexed copper ion emerging in electrodeless copper plating process for printed circuit board. Aliquat 336 has been recently suggested as promising extractant for removal of negatively complexed copper ion [Juang et al., 1999].

In this paper, negatively complexed copper ion from water was removed by using PDSE with CLAs containing Aliquat 336 diluted with nonpolar solvent kerosene. The performance of PDSE over conventional solvent extraction using straight Aliquat 336 was estimated. The effects of the type of water-soluble surfactants, concentration of surfactant Sodium Dodecyl Benzene Sulfonate (SDBS), Phase volume ratio (PVR) on PDSE of negatively complexed copper ion were investigated. In addition, the effect of anionic surfactant on back extraction in PDSE was also investigated.

Stability of CLAs in the continuous phase is an important factor

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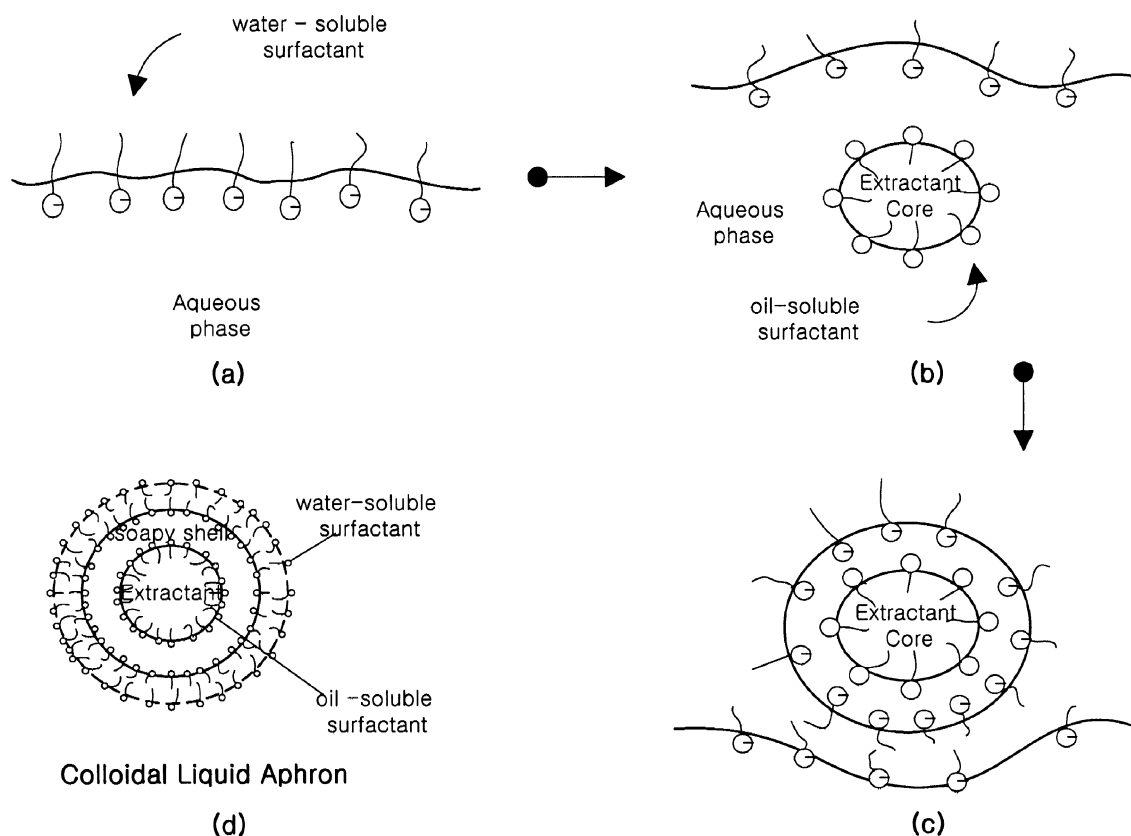


Fig. 1. Structure of an Aphron and formation of an Aphron (a) water-soluble surfactant is dissolved. (b) Extractant core with oil-soluble surfactant is introduced. (c) Extractant core will penetrate the surface by lifting up thin film of water that will then enshroud extractant core. (d) Structure of an Aphron [Sebba, 1986].

in PDSE. For example, the stability of CLAs in the continuous phase may influence mass transfer from pregnant phase to oil-core and be used for optimal recovery of CLAs from pregnant phase. CLAs in the continuous phase can be destroyed by various mechanisms. Break-up of CLAs is defined as macroscopic phase separation of CLAs in the continuous phase. Primarily, this arises from flocculation and coalescence of CLAs. Coalescence of CLAs is the joining of two (or more) aphrons to form a single aphron of greater volume. This irreversible process reduces interfacial area of CLAs. Flocculation of CLAs is to the mutual attachment of individual liquid aphrons to form loose assemblies of particles in which the identity of each is maintained, a condition that clearly differentiates it from the action of coalescence [Lye and Stuckey, 1998; Scarpello and Stuckey, 1999; Lee et al., 2002; Hahm et al., 2001]. Previous investigation on the stability of Aliquat 336 was conducted on the effect of process parameters on the stability of dilute dispersion of nonionically-stabilized CLAs containing Aliquat 336 diluted with octanol [Scarpello and Stuckey, 1999]. However, study of the stability of anionically-stabilized CLAs containing Aliquat 336 diluted with kerosene has not been reported in the literature. Therefore, the stability of anionically-stabilized CLAs containing Aliquat 336 diluted with kerosene was investigated in this work. The effect of pH on break-up rate of CLAs containing Aliquat 336 was also investigated.

## EXPERIMENTAL

### 1. Materials

EDTA (Ethylenediaminetetraacetic acid), the oil-soluble surfactant Tergitol 15-S-3, Hexadecyl Trimethyl Ammonium Bromide (HTAB), Polyoxyethylene sorbitan monooleate (Brij 35) were obtained from Sigma. The quaternary ammonium salt Aliquat 336, kerosene,  $\text{CuSO}_4$ , Anionic surfactant Sodium Dodecyl Benzene Sulfonate (SDBS), NaOH,  $\text{H}_2\text{SO}_4$  were purchased from Aldrich.

### 2. The Extraction Equilibrium in Conventional Solvent Extraction

EDTA-bearing  $\text{CuSO}_4$  solution that Cu(II) was complexed with EDTA of equal molar ratio was prepared. For conventional solvent extraction, straight extractants of Aliquat 336 diluted with nonpolar kerosene were prepared. Each solution of 20 ml was put into vial and stirred with a magnetic bar in a constant temperature bath for 12 hr. After that, the samples were separated in a centrifuge with 4,000 rpm for 10 min. From the bottom of phase-separated sample, a portion of the pregnant phase was picked up. The concentration of copper was measured with atomic absorption spectrometer of HP (perkin-Elmer 3300). The concentration of Cu(II) in the organic phase was calculated from mass balance. The same experiment was performed according to the change of the concentration of Aliquat 336.

### 3. The Preparation of Polyaphrons and CLAs

Polyaphrons were prepared by gradually introducing organic phase that contained Aliquat 336 diluted with kerosene and oil-soluble surfactant (0.01% v/v) into the aqueous phase with water-soluble

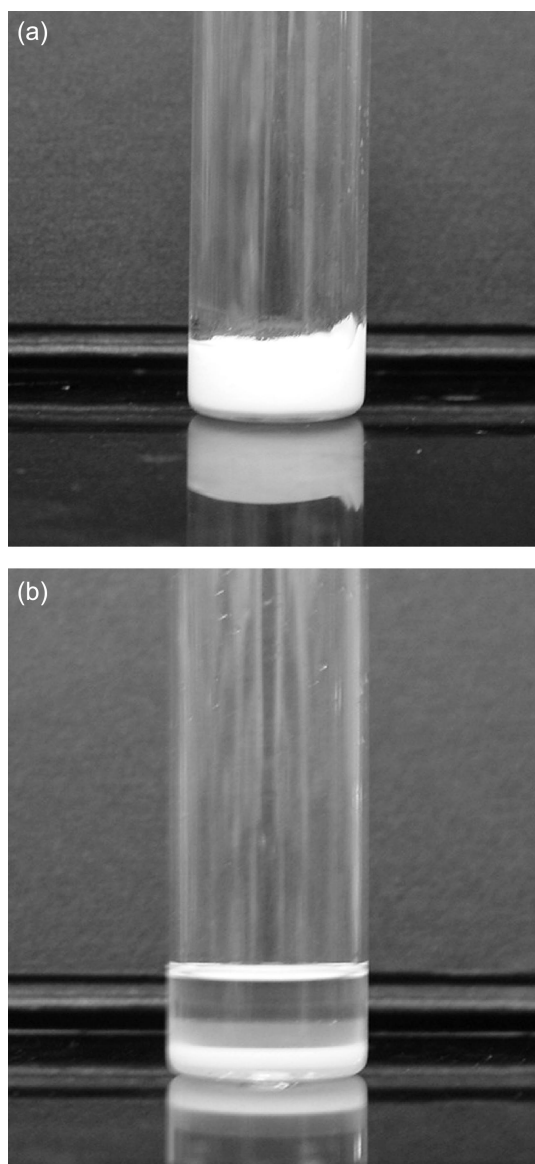


Fig. 2. (a) Stable polyaphrons: white creaming appearance. (b) Broken polyaphrons: phase separation has occurred.

surfactant. Stable polyaphrons show white creamy appearance and do not cause phase separation for a long time. CLAs were formed by dispersion of polyaphrons into the continuous phase. These polyaphrons are characterized into phase volume ratio (PVR).

$$PVR = \frac{\text{Dispersed solvent phase volume}}{\text{Continuous phase volume}} \quad (1)$$

Stable polyaphron and broken polyaphron are shown in Fig. 2

#### 4. Experimental Procedures in PDSE

In PDSE experiments, 10 ml of polyaphrons was added to 10 ml of EDTA-bearing  $\text{CuSO}_4$  solution in vial. The mixture was stirred in constant temperature bath for 30 min. After phase separation in a centrifuge, copper ion in the pregnant phase was analyzed with atomic absorption spectrometer. For the calculation of performance of PDSE over conventional solvent extraction, PDSE experiments were conducted with the same condition of conventional solvent extraction experiments.

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#### 5. Break-up Measurement of CLAs

A method to measure emulsion stability was applied for the measurement of break-up measurement of CLAs [Kang et al., 2000]. Prepared polyaphrons were transferred to a measuring cylinder. In order to produce CLAs, the continuous phase as destabilizing agent was gradually added to a measuring cylinder containing polyaphrons. The break-up of CLAs in the continuous phase arises from the formation of a film at the amine extractant-continuous phase interface. Amine solvent volume released to surface was recorded against time.

### RESULTS AND DISCUSSION

#### 1. The Effect of Various Surfactants on PDSE

The effect of various surfactants on Removal (%) is shown in Fig. 3. Removal (%) that represents the extent of removing copper in PDSE is defined as follows:

$$\text{Removal (\%)} = \frac{C_{\text{preg, PDSE}}^b - C_{\text{preg, PDSE}}^a}{C_{\text{preg, PDSE}}^b} \times 100 \quad (2)$$

Where  $C_{\text{preg, PDSE}}^b$ ,  $C_{\text{preg, PDSE}}^a$  is the concentration of copper in the preg-

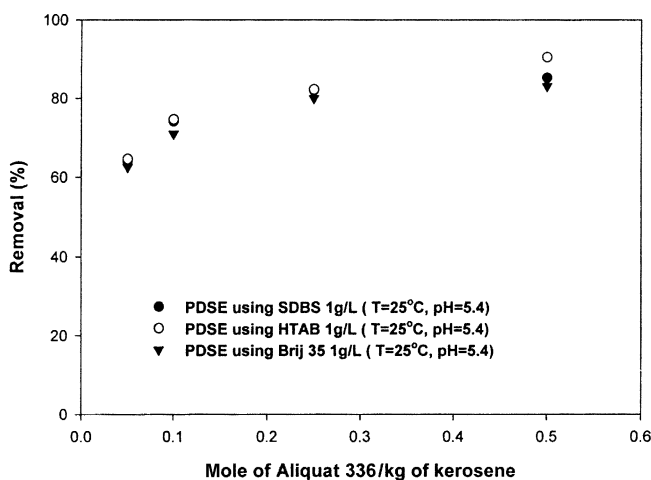


Fig. 3. Effect of surfactants type on removal (%) in PDSE using Aliquat 336.

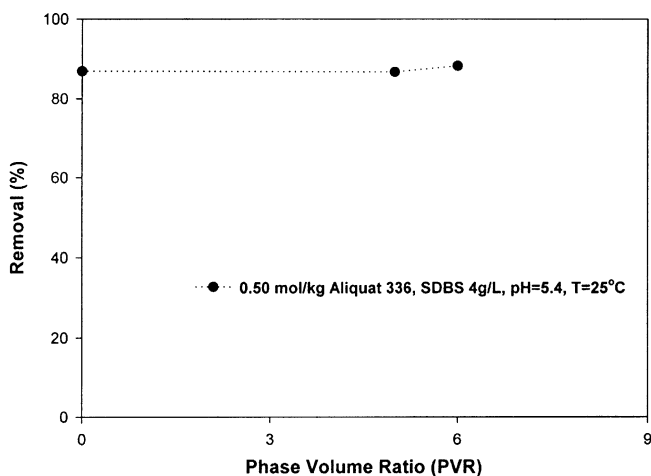


Fig. 4. Effect of PVR on PDSE.

nant phase before and after PDSE, respectively. Water-soluble surfactants used were cationic HTAB, anionic SDBS, non-ionic Brij 35 and amine organic solvents used were Aliquat 336 diluted with nonpolar solvent kerosene. The effect of the type of surfactants on Removal (%) is small. In the following study, anionic SDBS was selected for PDSE.

## 2. The Effect of PVR on PDSE

Fig. 4 shows the effect of PVR on removal in PDSE. 4 g/L SDBS and 0.50 mol/kg Aliquat 336 diluted with nonpolar kerosene were used for manufacture of polyaphrons. Maximum PVR obtained is 6 and the effect of PVR on removal is negligible. Therefore, PVR=5 was used in the further study.

## 3. Mass Transfer Rate Enhancement in PDSE

The major advantage of PDSE over conventional solvent extraction is the enhancement of mass transfer rate. The mass transfer rate in solvent extraction can be described as follows [Scravastava et al., 2000; Lye and Stuckey, 2001].

$$R = K_o \times A \times (\text{driving force}) \quad (3)$$

Where  $K_o$  is the overall mass transfer coefficient and  $A$  is interfacial area per unit volume. The useful approach to increase mass transfer rate in the extraction process is to increase the interfacial area. Mechanical mixing is usually used in conventional solvent extraction for increase of interfacial area. But this method leads to considerable mixing energy cost. On the other hand, the value of  $A$  in PDSE can be increased by comminuting extractants with two kinds of surfactants prior to the mixing with the pregnant phase. So, mass transfer rate can be enhanced by a large increase of interfacial area. In addition, mixing energy saving in PDSE can be accomplished by comminuting only one organic phase instead of both pregnant phase and organic phase as in conventional solvent extraction. Figs. 5 and 6 show this mass transfer rate enhancement in PDSE over conventional solvent extraction. The performance of PDSE over conventional solvent extraction is characterized as  $E_0$ .

$$E_0 = \frac{C_{preg,CSE}^b - C_{preg,PDSE}^b}{C_{preg,CSE}^b} \quad (4)$$

Where  $C_{preg,PDSE}^b$  is the concentration of copper in the pregnant phase

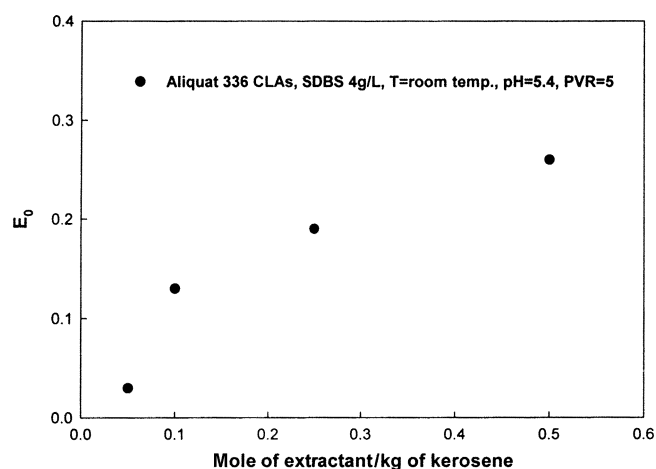


Fig. 5.  $E_0$  obtained according to Aliquat 336 concentration without mixing; settling time; 30 min.

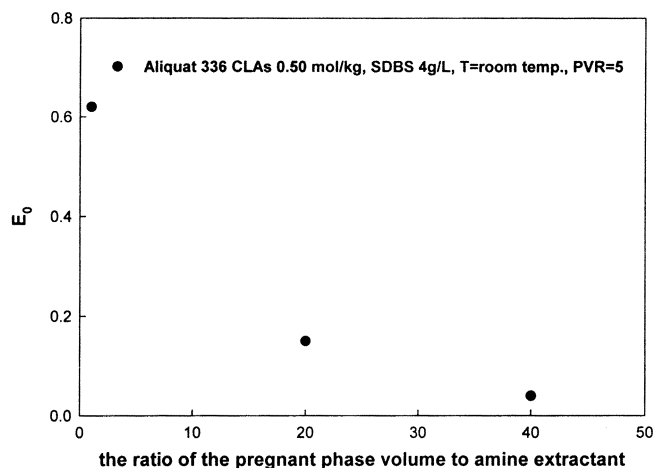


Fig. 6.  $E_0$  obtained according to the ratio of the pregnant phase volume to amine extractant without mixing; settling time; 30 min.

after PDSE  $C_{preg,CSE}^b$  and is the concentration of copper in the pregnant phase after conventional solvent extraction. When  $E_0$  becomes zero, mass transfer rate of PDSE is equal to that of conventional solvent extraction.  $E_0$  above zero means that PDSE has higher mass transfer rate than conventional solvent extraction. The pregnant phase volume and CLAs or straight extractant were contacted and settled in a separatory funnel for 30 min. without mechanical mixing. Absence of mechanical mixing is able to compare mass transfer rate between PDSE and conventional solvent extraction. Detailed experimental conditions were room temperature, pH=5.4, SDBS 4 g/L and PVR=5. Fig. 5 shows mass transfer rate enhancement of PDSE according to the change of concentration of Aliquat 336. Fig. 6 shows the effect of the ratio of the pregnant phase volume to amine extractant on  $E_0$ . As the ratio of the pregnant phase volume to amine extractant changed, PDSE and conventional solvent extraction proceeded in a separatory funnel for 30 min without mechanical mixing. The concentration of Aliquat 336 was 0.50 mol/kg. We can see that mass transfer rate is enhanced below about 40 in PDSE.

## 4. The Effect of Concentration of Surfactant SDBS on PDSE

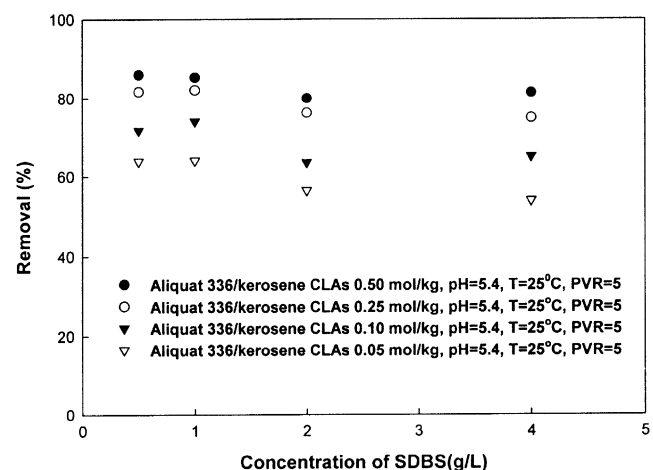


Fig. 7. The effect of concentration of surfactant SDBS on removal (%) in PDSE. Mixing time with a magnetic bar; 30 min and PVR=5.

The effect of the concentration of SDBS in the aqueous phase on solute transfer is shown in Fig. 7. 10 ml of polyaphrons of Aliquat 336 were mixed with 10 ml of the pregnant phase for 30 min. The pregnant phase for Aliquat 336 had negative complexed copper ion with EDTA. PDSE was preceded with changing the concentration of SDBS. In PDSE using Aliquat 336 CLAs, the fact that Removal (%) slightly decreases with rising the concentration of SDBS under the condition of constant concentration of Aliquat 336 means SDBS has no significant effect on solute transfer. Small decrease of Removal (%) results from repulsive interaction between anionic surfactant SDBS and negative EDTA-bearing copper ion. The effect of concentration of Aliquat 336 under constant concentration of SDBS is also shown in Fig. 7. Removal (%) increases with increasing Aliquat 336 concentration at constant concentration of SDBS. If PDSE using Aliquat 336 CLAs were affected by SDBS, Removal (%) would not be changed under constant concentration of SDBS. Therefore, the effect of concentration of surfactant SDBS on solute transfer in case of Aliquat 336 CLAs is negligible and only Aliquat 336 is concerned with transfer of copper from the pregnant phase to Aliquat 336-core in case of PDSE with Aliquat 336 CLAs.

### 5. The Comparison of the Quantity of Copper Transferred to Aliquat 336 Core in PDSE and Conventional Solvent Extraction

In section 3.4, PDSE was found to be not influenced by anionic SDBS. But comminuting straight amine extractants by using surfactants may influence solute transfer between Aliquat 336 and complexed copper ion. Fig. 8 shows this influence. Polyaphrons with PVR=5 were prepared by using anionic surfactant SDBS of 4 g/L in the aqueous phase. Both PDSE and conventional solvent extraction were performed with being mixed to reach equilibrium condition with a magnetic bar for 12 hr. To obtain data, the concentration of copper ion in the pregnant phase in PDSE was measured in the same experimental condition as conventional solvent extraction by using straight amine extractant. The amount transferred to amine core is nearly the same. This means that the increase of the amount of solute transfer in PDSE using Aliquat 336 CLAs in comparison with conventional solvent extraction under constant concentration of SDBS does not occur. Since PDSE is not influenced by SDBS,

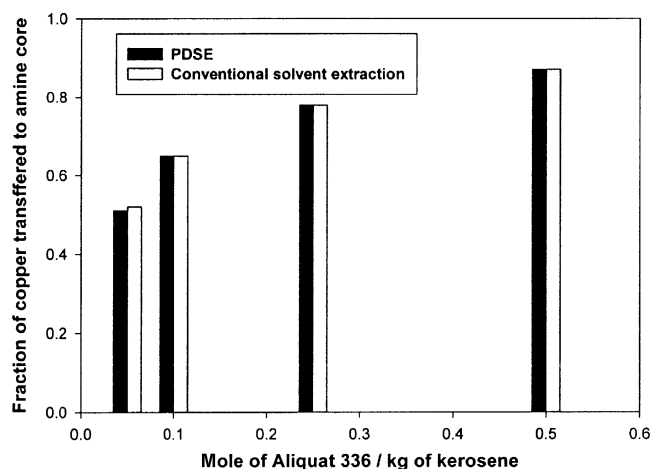


Fig. 8. Comparison of the amount of copper transferred to amine core in PDSE and conventional solvent extraction.

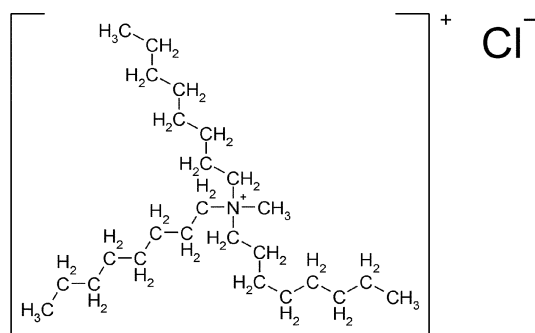
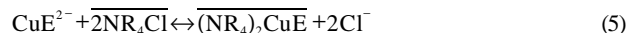


Fig. 9. Chemical structure of Aliquat 336.

comminuting straight amine extractant into micro-sized CLAs does not influence copper transfer. This result may be interpreted as follows. Extraction of negatively complexed copper using Aliquat 336 occurs by the exchange between complexed copper ion and  $\text{Cl}^-$  of Aliquat 336 according to following equation [Juang et al., 1999].



Where,  $\text{NR}_4\text{Cl}$  is Aliquat 336, E is EDTA.

As Fig. 9 shows, comminuting straight Aliquat 336 does not influence the exchange of chloride ion with complexed-copper ion because quaternary ammonium salt is forming ionic bond with chloride ion without steric hindrance by long chain alkyl group. Therefore, the increase of the amount of solute transfer in PDSE using Aliquat 336 CLAs in comparison with conventional solvent extraction may be negligible.

### 6. Back Extraction of Negatively Complexed Copper from Aliquat 336 Used

Eq. (5) is used to recover pure Aliquat 336 from Aliquat 336 loaded with copper. For the reverse reaction, NaCl solution was added into 0.5 mol/kg Aliquat 336 loaded with copper. Efficiency of recovery is defined as follows:

$$\text{Recovery (\%)} = \frac{Y}{Y_0} \times 100 \quad (6)$$

where, Y is concentration of copper in the pregnant phase after back extraction and  $Y_0$  is initial concentration of copper in the pregnant phase before PDSE.

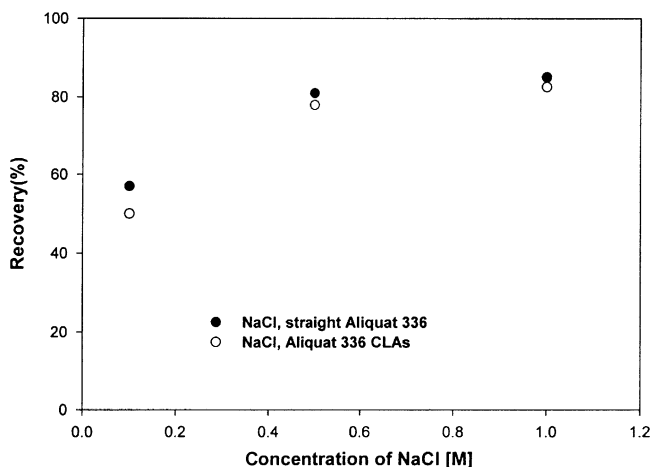


Fig. 10. Back extraction of copper from Aliquat 336 used.

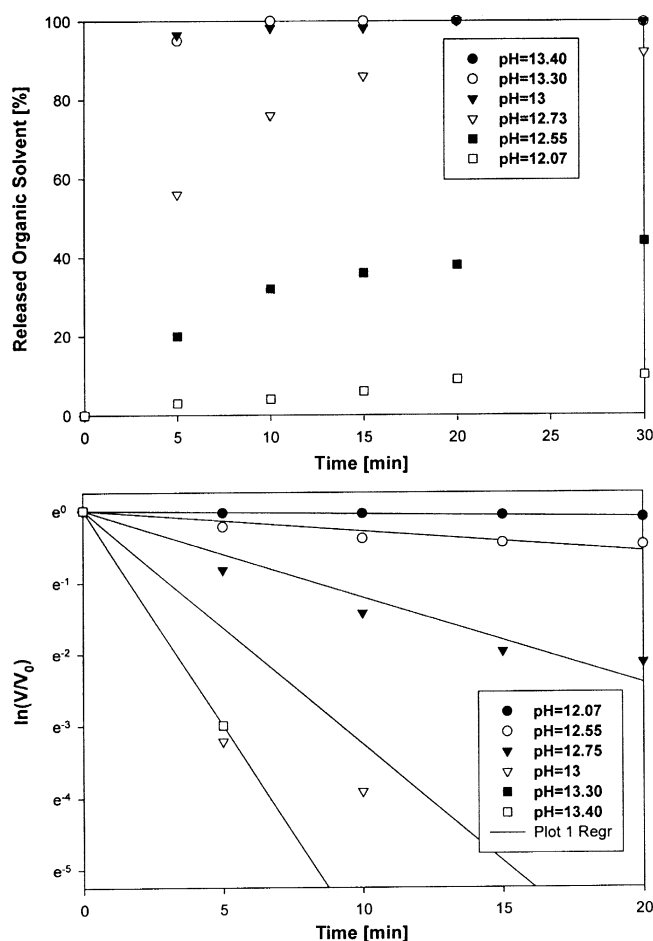


Fig. 11. (a) Effect of  $\text{OH}^-$  on stability of CLAs containing Aliquat 336 in the continuous phase. (b) pseudo-first-order plot obtained.

Fig. 10 shows recovery of copper ion by back extraction. As concentration of NaCl increases, Recovery (%) also increases in both straight Aliquat 336 which does not use surfactants and Aliquat 336 CLAs. In addition, difference of Recovery (%) between straight Aliquat 336 and Aliquat 336 CLAs is small. This means that recovery of organic amine solvent by using NaCl is not affected by surfactant SDBS.

### 7. The Effect of pH on Stability of CLAs

Figs. 11, 12 shows the effect of  $\text{OH}^-$ ,  $\text{H}^+$  on the stability of CLAs containing Aliquat 336 diluted with kerosene. Organic solvent released to the surface is plotted versus time at various concentration of  $\text{OH}^-$ ,  $\text{H}^+$  for CLAs containing 0.50 mol/kg Aliquat 336 diluted with kerosene. CLAs were prepared with anionic SDBS 4g/L, PVR= 5. The ratio of CLAs to continuous phase is 5: 40 ml. Stability of

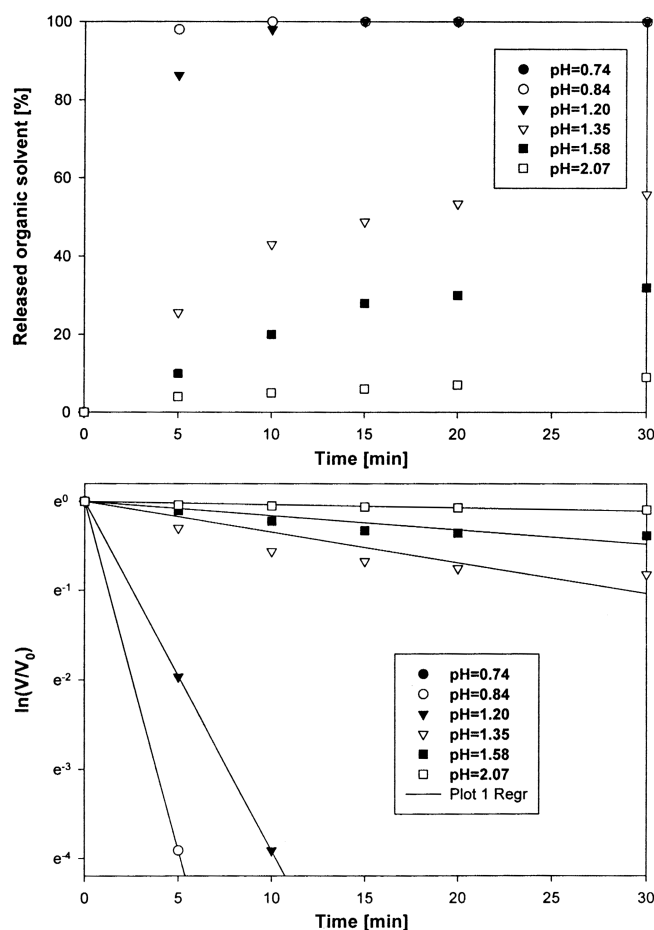


Fig. 12. (a) Effect of  $\text{H}^+$  on stability of CLAs containing Aliquat 336 in the continuous phase. (b) pseudo-first-order plot obtained.

CLAs decreases with increasing concentration of  $\text{OH}^-$ ,  $\text{H}^+$ . Break-up of CLAs mainly arises from destabilization of CLAs through coalescence. The kinetics of the coalescence process of CLAs was proposed to follow pseudo-first-order reaction kinetics, namely,

$$\frac{V(t)}{V_0} = \exp(-kt) \quad (7)$$

where  $V(t)$  is the remaining volume of CLAs,  $k$  is a first order rate constant, and  $t$  is the time elapsed. From Eq. (7), a first order half-life can be calculated from, [Lye and Stuckey, 1998]

$$t_{1/2} = \frac{\ln 2}{k} \quad (8)$$

Half-life obtained is summarized in Table 2. A plot of  $\ln(V/V_0)$  against

Table 1. The properties of surfactants

Surfactant	Molecular formula	MW/CMC (mM)	Charge
Sodium Dodecyl Benzene Sulfonate (SDBS)	$\text{C}_{12}\text{H}_{25}\text{O}_6\text{H}_4\text{SO}_3\text{Na}$	348.5/1.5	Anionic
Hexadecyl Trimethyl Ammonium Bromide (HTAB)	$\text{C}_{19}\text{H}_{42}\text{NBr}$	364.5/0.9	Cationic
Polyoxyethylene sorbitan momooleate (Brij 35)	$\text{C}_{12}\text{H}_{25}(\text{OCH}_2\text{CH}_2)_n\text{OH}$ $n=23$	1200/0.06	Nonionic
Tergitol 15-s-3	Oil soluble	336/NA	-

**Table 2. Half-life**

pH	$t_{1/2}$ [min]	pH	$t_{1/2}$ [min]
0.74	N/A <sup>a</sup>	12.07	146
0.84	N/A <sup>a</sup>	12.55	24.5
1.2	1.77	12.75	14.4
1.35	20.1	13	N/A <sup>a</sup>
1.58	43.3	13.3	N/A <sup>a</sup>
2.07	193	13.4	N/A <sup>a</sup>

<sup>a</sup>In this condition, break-up of CLAs do not follow pseudo first order reaction kinetics.

time is also shown in Figs. 11, 12. As expected in Eq. (8), straight lines are obtained. However, there are some concentrations that do not follow pseudo-first-order reaction kinetics. The founding of bad fit of pseudo-first order model means break-up of CLAs does not take place through coalescence. In other words, stripping of a thin aqueous soapy film, not coalescence of individual aphrons, may cause this deviation of pseudo first order reaction kinetics.

## CONCLUSIONS

In this work, separation characteristics of negatively complexed copper in PDSE and stability of anionically-stabilized Aliquat 336 CLAs diluted with kerosene were studied for development of PDSE process. The following conclusions were obtained.

1. In PDSE using Aliquat 336 CLAs, the effect of concentration of anionic SDBS on solute transfer and the effect of the surfactant type were negligible. Back extraction of negatively complexed copper was not disturbed by surfactant. Therefore, PDSE using Aliquat 336 CLA can be used for treatment of negatively complexed copper without the influence of surfactant. In addition, the effect of PVR on removal is negligible.

2. PDSE using Aliquat 336 CLAs made out of SDBS showed mass transfer rate enhancement in comparison with conventional solvent extraction using straight Aliquat 336 under experimental conditions without mechanical mixing.

3. Under experimental conditions with enough mechanical mixing, the amount of copper transferred to Aliquat 336 core from the pregnant phase was nearly similar in both PDSE using anionic SDBS and conventional solvent extraction.

4. Break-up of anionically stabilized CLAs containing Aliquat 336 diluted with kerosene follows pseudo-first-order model at low ionic strength. But, the pseudo-first-order model cannot be applied to regions of high concentration of  $H^+$ ,  $OH^-$ , indicating the break-up mechanism does not follow through coalescences.

## ACKNOWLEDGMENTS

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## NOMENCLATURE

$C_{preg, PDSE}^b$ : the concentration of copper in the pregnant phase before

PDSE [mg/L]

$C_{preg, PDSE}^a$ : the concentration of copper in the pregnant phase after PDSE [mg/L]

$C_{preg, CSE}^b$ : the concentration of copper in the pregnant phase before conventional solvent extraction [mg/L]

$E_0$ : the performance of PDSE over conventional solvent extraction

$t_{1/2}$ : half-life [min]

$t$ : time [min]

$R$ : volumetric mass transfer rate

$K_o$ : the overall mass transfer coefficient

$A$ : interfacial area per unit volume

$t_{fast}$  is half-life that stability of CLAs changes rapidly

$t_{slow}$  is half-life that stability of CLAs changes slowly

$Y$  is concentration of copper in the pregnant phase after back extraction

$Y_0$  is initial concentration of copper in the pregnant phase before PDSE

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