



Selective recovery of Au(III), Pt(IV), and Pd(II) from aqueous solutions by liquid–liquid extraction using ionic liquid Aliquat-336



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ABSTRACT

In this study, selective extraction and recovery of Au(III) from a tertiary metal solution containing Au(III), Pt(IV), and Pd(II) were investigated using ionic liquid Aliquat-336. The effects of Aliquat-336 concentration, solution pH, extraction time, and initial multi-metal concentration were examined in details. From the extraction efficiency and selectivity coefficient (α_{sel}), it was noted that Aliquat-336 was a very fast and effective extractant for selective extraction of Au(III) from the multi-metal solutions. Through sequential extraction, high purity of each metal solution was separately obtained from low and high concentrations of multi-metal solutions. Therefore, the present study suggests a new way to separate and recover precious metals of high purities using ionic liquids.

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1. Introduction

In recent years, precious metals (PMs) including gold, platinum, palladium, silver, and rhodium have been widely used in many industrial areas such as jewelry, corrosion-resistant materials, chemical catalysts, and various chemical processes, owing to their specific properties such as lustrous, ductile, noncorrosive and high stability [1–5]. However, the limited resource and endless demand of PMs in industrial spheres have led to the urgent recovery of these noble metals from related PMs-loaded wastewaters. A variety of technologies such as solvent extraction [6,7], adsorption [8,9], ion exchange [10], precipitation [11], and electrowinning [12] have been extensively used in the recovery of PMs from their aqueous effluents. Among them, the solvent extraction process has been receiving considerable attention and eliciting widespread interest in the past few decades owing to its fast extraction rate and high loading property [7,13–17]. Nonetheless, the process has a difficulty of simultaneously separating and purifying PMs from multi-metal mixtures owing to their similar physical and chemical properties [1,18]. Thus, developing effective extraction processes for the recovery and separation of PMs to obtain high purity metal solutions is necessary.

A key point for the effective application of the solvent extraction for selective recovery of PMs is to find an appropriate extractant and to design suitable separation procedures. For this purpose, ionic liquids (ILs) have recently attracted attentions instead of traditional extractants for

recovery of metals owing to their specific characteristics such as negligible vapor pressures with high thermal stabilities and tunable viscosities, and their good properties towards metals [4,15,19]. For example, a model IL Aliquat-336, tricaprylylmethylammonium chloride, (a hydrophobic quaternary ammonium salt ($[R_3NCH_3]^+ Cl^-$)) showed good extraction capacity for metals. Nonetheless, most studies focused on the single metal extraction by using Aliquat-336 [20–25]. It must be noted that in some studies Aliquat-336 had been combined with other extractants in order to separate PMs [14]. However, the more extractants use, the higher separation cost is. To date, less effort was made to selectively extract and separate targets PMs solely by using Aliquat-336.

In this study, therefore, IL Aliquat-336 was chosen as a model extractant and its selective extraction performance was investigated in a ternary-metal solution containing Au(III), Pt(IV), and Pd(II). Furthermore, an effective sequential extraction process only with one extractant of Aliquat-336 was successfully designed to separate Au(III)/Pt(IV)/Pd(II) and obtain high purity of each metal solution. Therefore, the present study suggested a new possibility to separate Au(III), Pt(IV), and Pd(II) using appropriate Aliquat-336 concentrations through the designed flow sheet.

2. Materials and methods

2.1. Materials

Multi-metal solutions were prepared by dissolving certain amounts of $H_2PtCl_6 \cdot 5.5H_2O$ (Kojima Chem., Korea), $PdCl_2$ (Kojima Chem., Korea) and $HAuCl_4 \cdot 3.6H_2O$ (Kojima Chem., Korea) in 0.1 M hydrochloric acid.

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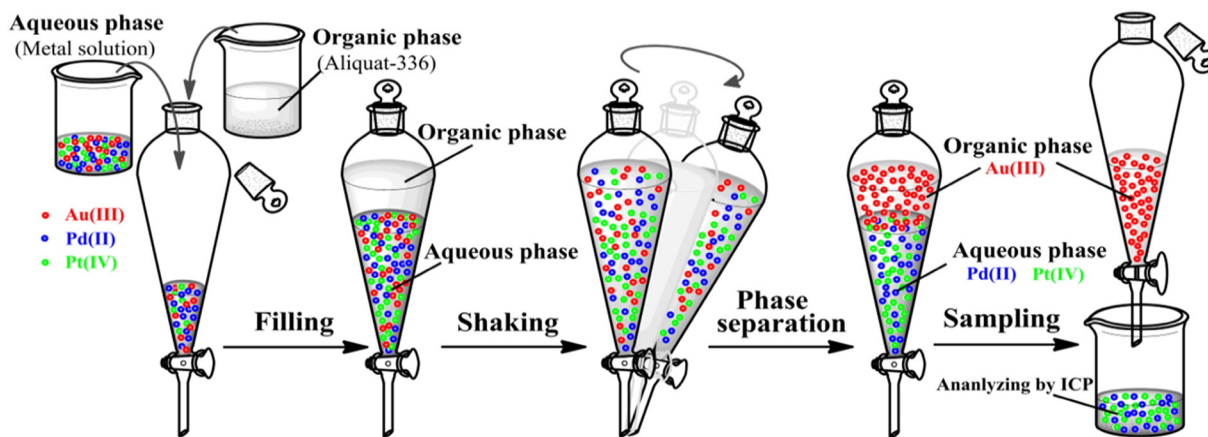


Fig. 1. Aliquat-336 structure (a) and the extraction process (b).

Aliquat-336 (Sigma-Aldrich, Korea) and benzene (Samchun Pure Chem., Korea) were used as the solvent extractant and diluent, respectively. All other chemicals were of analytical grades, and purified water supplied by deionized water equipment (Millipore/direct-Q3UV, USA) was used throughout the experiments.

2.2. Liquid–liquid extraction procedure

To evaluate the extraction efficiencies of metals from aqueous phase, benzene was used as the organic phase owing to its hydrophobic property and good solubility for Aliquat-336. The extraction procedure is as follows: equal volumes of each phase solution (40 mL) were filled in a separation funnel and vigorously shaken to make an environment for the phase transfer of the metals between the aqueous (Au(III)/Pt(IV)/Pd(II) mixture solution) and organic (Aliquat-336 + benzene) phases at room temperature within appropriate time. After the phase separation, the system was stabilized, and samples were taken from the aqueous phase. Then, metal concentrations in the aqueous phase were measured by inductively coupled plasma-atomic emission spectrometry (ICP-AES) after proper dilutions. The extraction process is shown in Fig. 1. The extraction efficiency (E), distribution coefficient (K_d), and selectivity coefficient (α_{sel}) were calculated using the following equations:

$$E = \frac{(C_{aqi} - C_{aqf})}{C_{aqi}} \quad (1)$$

$$K_d = \frac{(C_{aqi} - C_{aqf})}{C_{aqf}} \quad (2)$$

$$\alpha_{sel} = \frac{K_{dMI}}{K_{dMII}} \quad (3)$$

where E , K_d , and α_{sel} are the extraction efficiency, distribution coefficient, and selectivity coefficient, respectively. C_{aqi} and C_{aqf} are the initial and final metal concentrations in aqueous phase, respectively. MI and MII are metal types.

2.3. Stripping process

After moving the metals from aqueous to organic phase, a stripping step was performed for removing the metals existing in the organic phase to aqueous phase for recovery. The stripping experiments followed same procedure as the extraction experiments. Several types of reagents were tested to check their stripping capacities. After first extraction step, 40 mL of each loaded organic phase was separated and

mixed with newly prepared 40 mL of an aqueous solution containing the stripping reagents for 5 min. Here, the time effect for the stripping process was investigated and the results showed that the stripping efficiencies of 5 and 10 min were same (data not shown). Thus, 5 min was chosen for the stripping process. After the phase separation, the metal concentrations in the aqueous phases were measured by ICP-AES.

3. Results and discussion

3.1. Factors affecting the selective extraction of Au(III)

For optimizing the selective extraction process, the effects of parameters such as phase contact time, pH, concentration of Aliquat-336, and initial metal concentration were investigated. The extraction efficiency (E) and selectivity coefficient (α_{sel}) were used as the measures of the relative fitness of selective performance towards Au(III), and the results are discussed as follows.

3.1.1. Effect of phase contact time

The extraction behavior of Au(III)/Pt(IV)/Pd(II) (200 mg/L each) prepared in hydrochloric acid (0.1 M) solution with 0.6 g/L Aliquat-336 in benzene was investigated at varied times from 0 to 30 min. Fig. 2(a) shows that the Au(III) extraction efficiency reached >99% within 0.5 min, and after that the extraction remained almost constant. Moreover, the extent of the extraction of Pt(IV) and Pd(II) during 30 min were <5 and 7%, respectively. These results indicate that Aliquat-336 enables fast extraction of Au(III) compared to Pt(IV) and Pd(II). Notably, Aliquat-336 is a good surfactant, and this fast extraction performance may be partially because of decreasing interfacial tension [19,26,27].

Furthermore, the estimated metal selectivity coefficients (α_{sel}) as a function of time are also shown in Fig. 2(b). Due to stable extraction capacity of Aliquat-336 towards Au(III), Pt(IV) and Pd(II), the selectivity coefficients of $\alpha_{Au/Pt}$ and $\alpha_{Au/Pd}$ almost remained constant without significant changes. In consideration of sufficient extraction time, 5 min was chosen as a contact time for the following selective extraction experiments.

3.1.2. Effect of pH

Fig. 3 shows the effect of pH on the selective extraction of Au(III) from the ternary metal solutions. The extraction experiments were conducted in the pH range between 1 and 5 to avoid precipitation of the studied metals [28]. The extraction efficiency of Au(III) reached ~100% over the entire tested pH range (Fig. 3(a)). In the cases of Pt(IV) and Pd(II), the extraction percentages increased from 3.51 to 6.10% and

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