

다양한 용액으로부터 용매추출에 의한 금과 은의 분리

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Separation of Gold and Silver from Diverse Solutions by Solvent Extraction

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요 약

금과 은은 귀금속으로 첨단소재를 제조하는데 사용된다. 용매추출은 다양한 침출용액으로부터 순수한 금과 은을 회수할 수 있는 중요한 공정이다. 본 논문에서는 cyanide, thiocyanate, thiosulfate, thiourea와 염산용액에서 금(I, III)과 은(I)의 용액화학 및 용매추출에 의한 분리를 고찰했다. 여러 단독 및 혼합추출제에 의한 금(I, III)과 은(I)의 용매추출 및 분리거동을 각 침출용액에서 추출반응과 추출제의 선택도를 토대로 비교했다. 염산용액이 용매추출에 의한 금과 은의 분리의 효율측면에서 적당하다.

주제어 : 금, 은, 수용액 화학, 용매추출, 분리

Abstract

Solvent extraction is an important process to recover pure gold and silver from various leaching solutions. The present work reviews the aqueous chemistry and solvent extraction separation of gold (I, III) and silver (I) from several leaching systems such as cyanide, thiocyanate, thiosulfate, thiourea and chloride medium. The extraction and separation behavior of gold (I, III) and silver (I) by various single and mixtures was compared on the basis of extraction reaction and the selectivity from these mediums. The chloride medium is recommended for the separation of gold and silver by solvent extraction in terms of extraction and stripping efficiency.

Key words : gold, silver, aqueous chemistry, solvent extraction, separation

1. INTRODUCTION

Gold and silver are precious metals with excellent chemical and physical properties. Therefore, these two metals are widely used in various industrial fields such

as jewelry, electronic and electrical devices, communication, and automobiles. Since the reserves for these metals are limited, recovery of pure gold and silver from spent resources has attracted much attention. Although gold can be selectively dissolved in NaCN solution, strict

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control of the reaction atmosphere renders NaCN less acceptable as a lixiviant. Roasting followed by leaching is widely employed to dissolve the gold and silver present in the spent resources. Once these precious metals together with other base metals are dissolved in the leaching solution, separation of gold and silver from the leaching solution is of importance in recovering precious metals with high purity. For this purpose, some separation methods, such as ion exchange (IX)¹⁻⁸, solvent extraction (SX)⁹⁻¹³, electrowinning¹⁴⁻¹⁸ and precipitation¹⁹⁻²¹ have been extensively studied. Among the above-mentioned separation methods, solvent extraction is regarded as a commercial process which can be applied for the separation of precious metals and base metals. Solvent extraction has some advantages over ion exchange in terms of loading capacity, the concentration range of the metals in the solution, and reaction kinetics²².

This review is aiming to summarize the solvent extraction process for the separation of gold and silver in different aqueous media. The reaction details and separation efficiency of several proposed processes have been reviewed.

2. AQUEOUS CHEMISTRY OF GOLD AND SILVER

Information on the aqueous chemistry of the metals is of importance in selecting an appropriate extractant for the extraction and separation of the metals. In the leaching of gold and silver present in diverse spent resources, several leaching reagents, such as cyanide, thiosulfate, thiourea and chloride are widely employed. Most of the reaction occurring in the aqueous phase can be considered as an acid-base reaction. According to HSAB theory, a soft acid has a strong tendency to

react with a soft base, while a strong acid reacts strongly with a strong base. Among the several leaching agents, cyanide (CN⁻), thiourea (NH₂), thiocyanate (SCN⁻), and thiosulfate (S₂O₃²⁻) are classified as soft bases. Therefore, Au(I) and Ag(I) which belong to soft acid have a strong tendency to form complexes with these leaching agents. However, Au (III) is classified as a hard acid and thus prefers to form complexes with hard base ligands like chloride ions²². Although Au(II) species has been identified in some solution, this species is unstable and thus is not considered in this review²³. Since the stability constants for the formation of the complexes reflects the degree of complex formation, the aqueous chemistry of Au(I), Au(III) and Ag(I) is discussed on the basis of these data in each medium.

2.1. Cyanide medium

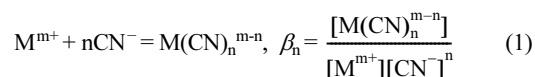
In cyanide leaching solution, the soluble complex of Au(I) is only Au(CN)₂⁻, while Ag(CN)₂⁻, Ag(CN)₃²⁻ and Ag(CN)₄³⁻ are known to exist as Ag(I) complexes^{1,24}. Therefore, the distribution of Ag(I) complexes depend on the cyanide concentration and solution pH. Au(III) has a stronger tendency to form complex with CN⁻ than Au(I)²⁴. As an alternative to cyanide, thiocyanate is widely employed as a leaching agent for gold and silver and has a strong affinity for gold and silver ions. Jinshan Li et al.²⁵ investigated the aqueous chemistry of gold and silver in the thiocyanate solution. When thiocyanate concentration is above 0.24 M and the initial concentration of Ag(I) is 20 mg/L, the predominant species of Ag(I) is found to be Ag(SCN)₃²⁻. As the initial concentration of Ag(I) increases, Ag(SCN)₄³⁻ and Ag (SCN)₃²⁻ can be formed in the solution.

The stability constants for the formation of gold and silver complexes with CN⁻ and SCN⁻ are listed in Tables

Table 1. Stability constants for the formation of gold and silver complexes with either cyanide or thiocyanate at 25°C

Stability constants	Au(I)		Au(III)		Ag(I)	
	CN ⁻	SCN ⁻	CN ⁻	SCN ⁻	CN ⁻	SCN ⁻
β ₂	2 × 10 ³⁸	1.47 × 10 ¹⁹	-	-	10 ^{20.9}	2.0 × 10 ⁸
β ₃	-	-	-	-	10 ^{21.8}	3.2 × 10 ⁹
β ₄	-	-	10 ⁵⁶	4.57 × 10 ⁴³	-	5.66 × 10 ⁹

1 and 2²⁶). In these tables, the stability constants are the equilibrium constants for the complex formation reaction represented in Eq. (1).



where M^{x+} represents Au or Ag.

2.2. Thiosulfate medium

Thiosulfate has some advantages as a leaching agent such as less environmental burden, high selectivity and less corrosive nature²⁷⁻³⁰). Therefore, some works have been done on the nature of the complexes of Au and Ag with thiosulfate. J. G. Webster³¹) reported that the most stable complex of Au(I) in neutral or alkaline solutions is $Au(S_2O_3)_2^{3-}$, while $Ag(S_2O_3)_2^{3-}$ exists in the moderately oxidizing conditions. Especially, Au and Ag can form complex (Au, Ag) $(S_2O_3)_2^{3-}$. However, during Au leaching by thiosulfate in the presence of some catalysts, the metallic Au could only be oxidized to Au(I)^{32,33}). As the oxidation-reduction potential of the solutions increased, thiosulfate can be oxidized to sulfate and thus $Ag(S_2O_3)_2^{3-}$ can be easily transformed into $AgS_2O_3^{-}$, Ag^{+} and $AgSO_4^{-}$. The equilibrium constants for the formation of complexes with thiosulfate are listed in Table 2.

2.3. Thiourea medium

Thiourea can form stable cationic complexes with gold and silver in aqueous solutions. $Au[CS(NH_2)_2]^{+}$ ($\beta_2 = 2.0 \times 10^{21}$) exists as a stable complex between Au(I) and thiourea, while no stable complex has been known for Au(III) owing to its reduction to Au(I) by thiourea³⁰). On the contrary, Ag(I) can form several complexes with thiourea. One complex of Ag(I) is $Ag[CS(NH_2)_2]_3^{+}$ ($\beta_3 = 1.3 \times 10^{13}$). The presence of some ions like NH_4^{+} and Fe^{3+} would accelerate the formation of Ag(I) complexes with thiourea³⁴⁻³⁹).

2.4. Chloride medium

Chloride ion has a strong tendency to form stable complexes with gold and silver and thus is widely employed in the leaching process. For this purpose, either aqua regia or hydrochloric acid in the presence of some oxidizing agents are generally employed in the leaching of some refractory metals^{23,40}). The formation of complexes of gold and silver with chloride ion have an important influence on the solvent extraction. Unlike the other medium, $AuCl_4^{-}$ is the unique stable complex of gold in chloride medium. The order of the stability constants of Au(III) complexes $[AuX_2]^{-}$ is given as follows²³).



When the concentration of chloride ion is high, Ag(I) can form several complexes with chloride ion, such as $AgCl_{(aq)}$, $AgCl_2^{-}$, $AgCl_3^{2-}$ and $AgCl_4^{3-}$ at room temperature²⁷⁻³⁰). Although the solubility of silver in chloride medium depends on the concentration of chloride ion, the solubility of Ag(I) is less than 50 ppm⁴¹⁻⁴⁴). Some researchers also found that silver dihalo complexes exist in different organic solutions⁴⁵).

3. SEPARATION OF Au AND Ag FROM DIFFERENT AQUEOUS BY SOLVENT EXTRACTION

The nature of the extractants employed for the separation of gold and silver can be classified into three kinds. Therefore, acidic extractants (HA), solvating extractants (L) and amine-based extractants (R_3N) and their mixtures are going to be discussed in this review on the basis of their selectivity and extraction performance for the several mediums.

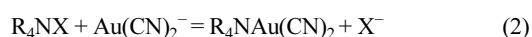
3.1. Cyanide media

3.1.1. Amine based extractants

Table 2. Equilibrium constants for the formation of thiosulfate complexes of gold and silver at 25°C

Complexes	$[Ag(S_2O_3)_2]^{3-}$	$[Au(S_2O_3)_2]^{3-}$	$[AuS_2O_3]^{-}$	$[AgS_2O_3]^{-}$
logK	13.83	26.8	10.4	8.82

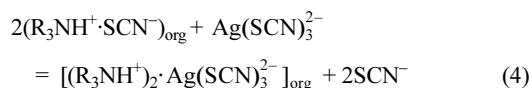
P. A. Riveros⁴⁶⁾ separated Au(I) from cyanide media by Aliquat 336 diluted in Solvesso 150 and Eq. (2) represents the extraction reaction. The diluent has an important influence on the extraction of Au(I), resulting in the massive emulsions formation. Since the concentration of Ag(I) was low, the extraction behavior of Ag(I) was not discussed.



where R_4NX represent ammonium salt of Aliquat 336 and X^- is common anion such as Cl^- or HSO_4^- .

Another amine-based extractants of N,N' -bis(2-ethylhexyl)guanidine were employed by G.A. Kordosky et al.⁴⁷⁾ to extract Ag(I) and Au(I) from cyanide leaching solution. Since amine should be protonated to take part in the reaction, adjustment of pH is important in the extraction of Au(I) and Ag(I). When solution pH is higher than 10.5, the extraction percentage of Au(I) decreased quickly. Moreover, the extractant's selectivity for Au(I) over Ag(I) is not high.

In thiocyanate solution, amine extractants such as Primene JMT and TOA are employed to extract Ag(I) for the determination of the reaction mechanism in the presence of ammonium by Sanuki et al.⁴⁸⁾. The reaction can be shown as Eqs. (3) and (4)



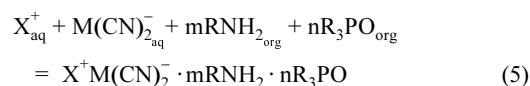
where R_3N refers the Primene JMT or TOA.

Xie et al.⁹⁾ reported that LIX 7950 could extract Au(I) and Ag(I) over other base metals such as Cu, Zn, Ni and Fe. The results showed the charge density of complexes has an important influence on the extraction and the extraction selectivity order is $Au > Ag > Zn > Ni > Cu > Fe$. However, the separation factor between Au(I) and Ag(I) is very small.

3.1.2. Mixture of extractants

Several extractant mixtures have been employed to separate Au(I) and Ag(I) from the cyanide solutions. F.J. Alguacil et al.⁴⁹⁾ investigated the extraction behavior

of Au(I) and Ag(I) over other base metals in the cyanide solution by the mixture of Primene JMT and Cyanex 921. Then F.J. Alguacil et al. further studied the mixture of Primene JMT and Cyanex 923⁵⁰⁾. The results indicated that the extraction order is $M(CN)_2^- > M(CN)_4^{n-} > M(CN)_6^{n-}$. The solvent extraction reaction can be expressed as Eq. (5).



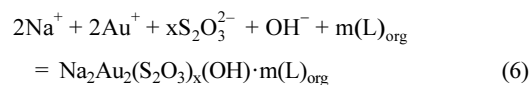
3.2. Thiosulfate medium

Many studies have been done on the extraction of gold(I) from thiosulfate solutions by amine extractants, neutral extractants and their mixtures such as N_{1923} , DNA, TOA, TRAO, TBPO and TBP^{39,51-53)}.

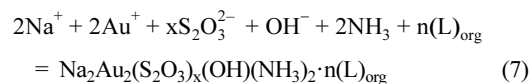
3.2.1. Solvating extractants

Compared to the cyanide medium, the species of $Au(S_2O_3)_2^{3-}$ has higher ionic charge and thus would be complexed with OH^- to decrease the charge density and then be extracted into the organic phase at $pH > 10^{39)}$. The results indicated that TBP is better than TBPO as an extractant for Au(I). Meanwhile, the presence of ammonia in the solution has an important influence on the extraction of gold from thiosulfate media. The solvent extraction reaction in the absence and presence of ammonia can be displayed as Eqs. (6) and (7).

In the absence of NH_3 ,



In the presence of NH_3 ,



where $1.5 < m < 2.5$, $x = 2-3$, $6 < n < 9$, L presents the neutral extractant.

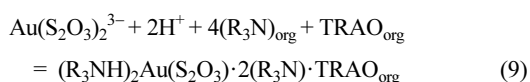
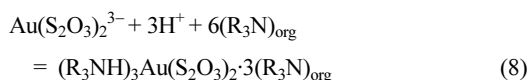
3.2.2. Amine based extractants

Aside from the extraction by neutral extractants, Zhao et al.⁵¹⁾ further compared the effect of different amine

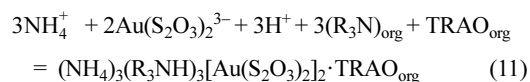
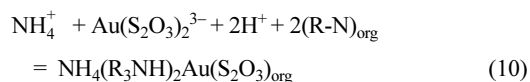
extractants, such as N₁₉₂₃, DNA and TOA on the extraction of Au(I) in the thiosulfate solutions. The extraction order among the amines is primary > secondary > tertiary amines, and the extraction percentage of gold(I) decreases as solution pH increases from 8 to 12. The extraction performance of the mixture of amine like N₁₉₂₃ and amine oxide TRAO were examined⁵³. The results suggested that addition of TRAO makes Au(I) extractable from weakly alkaline solution (pH = 6.5 ~ 8).

Since Au(S₂O₃)₂³⁻ can form complex with ammonia, the extraction reaction of Au(I) by the extractant mixtures in the absence and presence of ammonia can be implied as Eqs. (8) ~ (11).

In the absence of NH₃,



In the presence of NH₃,



where L, R₃N and TRAO refer to the neutral, amine extractants and amine oxide.

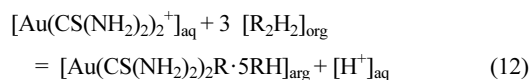
3.2.3. Mixture of extractants

Zhao et al.⁵² performed a series of experiments on the extraction of single Au(I) by using the mixture of neutral and amines extractant and reported that a mixture of N₁₉₂₃ and TBP is the most effective for the extraction of Au(I). When solution pH is less than 9, N₁₉₂₃ is considered as the main extractant and TBP works as a synergist during the extraction. When solution pH is higher than 9, the role of the extractants becomes the reverse. In addition, Zhao et al.⁵⁴ also worked to separate Au(I) from the leaching solution containing Ag(I) and other base metals by the mixture of N₁₉₂₃ and TRAO.

According to the results, the separation factor between Au(I) and Ag(I) becomes increased to 15 at pH lower than 8, compared to the low separation factor of 1.2 by single N₁₉₂₃.

3.3. Thiourea medium

Since the complexes of Au(I) and Ag(I) are cationic species in the acidic thiourea solution, cationic exchange reagents like D2EHPA has been employed for the separation of Au(I). Kim et al.⁵⁵ reported that the extraction reaction of Au(I) by D2EHPA can be revealed as Eq. (12)

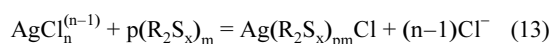


3.4. Chloride medium

Since the solubility of Ag(I) in chloride solution is very small, there are few works about the separation of Au(I) and Ag(I) from chloride solution by solvent extraction. Ag(I) is generally removed by precipitation before extraction⁴⁰. AgCl₂⁻ exists in strong chloride solution and thus should be considered in the course of refining of metals. Au(III), a hard acid has strong tendency to form stable complex with chloride ion, a hard base²². Therefore, AuCl₄⁻ is very stable in the chloride solution and the separation of Au(III) from other precious metals and base metals has been studied by many researchers^{10,56-59}.

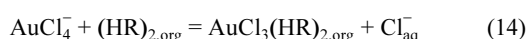
3.4.1. Acidic extractants

Compared to the cationic complexes of Ag(I) and Au(I) from thiosulfate and thiourea, most of Au(III) and Ag(I) exist as anionic complexes in chloride solution. Some acidic extractants such as Cyanex 302, Cyanex 301, SFI-6R and MSP-8 containing the functional groups of R - S or P = S have been employed for the extraction of Ag(I) from chloride solution. As HCl concentration increases to higher than 6 M, the extraction percentage of Ag(I) decreases and the extraction reaction can be represented as Eq. (13)⁴³:



where $\text{AgCl}_n^{(n-1)}$ refers to the complexes of Ag(I), p is the stoichiometric ratio; m is the degree of association; and $x = 1$; for Cyanex 301, $x = 2$.

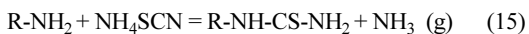
Meanwhile, Cyanex 302 was employed to separate Au(III) from Pd(II) and Pt(IV) as well as base metals containing Cu, Ni and Fe in chloride solution⁵⁶. However, the extraction percentage of Au(III) decreases as HCl concentration increases, which can be explained by the following Eq. (14).



3.4.2. Amine based extractants

Amine extractants have been employed for the separation of Au (III) from other precious metals except Ag together with base metals^{10-12,59-62}. Few works focused on the separation of Au(III) and Ag(I) from strong HCl solution. Ohto et al. examined the extraction behavior of Au(III) and Ag(I) by amide extractants from highly acidic chloride solution and found that the extraction behavior of Ag(I) depended on the nature of Ag(I) complexes with chloride ion⁶³.

Zuo et al.⁶⁴ reported that separation of either Au (III) or Ag(I) from chloride solution by the synthetic extractants from alkyl primary amine with ammonium thiocyanate (NH_4SCN). According to this work, Au (III) complex was extracted into the organic as $\text{AuCl}_3(\text{DTH})_3$ and only one species of Ag(I) was extracted but the formation of $\text{Ag}_m(\text{DTH})_n$ was not verified by this work. The extraction percentage of Au(III) and Ag(I) decreased with the increase of HCl concentration. The synthesis of extractants can be represented as Eq. (15).



4. Conclusions

The aqueous chemistry of gold and silver in the media of cyanide, thiosulfate, thiourea and chloride was reviewed, and the solvent extraction separation of gold and silver from the above media was discussed. From the review, the following conclusions were made.

1) In the cyanide systems, the complexes of Au(I) and Ag(I) are the main species. Owing to the toxicity

of cyanide, the extraction and separation behavior of Au(I) and Ag(I) was investigated by using solvating extractants but the selectivity for gold and silver is low.

2) Thiosulfate and thiourea medium are considered to be the promising reagents to recover gold and silver. Au(I) and Ag(I) could form stable complexes with thiosulfate and thiourea ions. Meanwhile, extraction selectivity of Ag(I) and Au(I) could be improved by addition of ammonium. In the case of thiourea system, there are few works on the solvent extraction of Au (I) and Ag(I) and cationic extractants of D2EHPA was used for the extraction.

3) In strong hydrochloric acid solution, the stable complex of Au(III) is AuCl_4^- , while Ag has low solubility but the nature of Ag(I) complex depends on the concentration of chloride ion. The extraction reaction of Au(III) and Ag(I) by cationic extractants containing $\text{R} - \text{S}$ and $\text{P} = \text{S}$ occurs through anion exchange. In the case of amine extractants, the extraction reaction of Ag(I) is complicated, whereas AuCl_4^- takes part in the reaction. Few works investigated the separation of Au(III) and Ag(I).

4) In terms of separation efficiency between gold and silver, chloride medium is recommended among cyanide, thiourea, thiosulfate and chloride medium.

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