

다양한 자원으로부터 은의 화학적 침출

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Chemical Leaching of Silver from Diverse Resources

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요 약

은은 특수한 물성을 지니고 있으며 첨단소재용 원료로 사용된다. 따라서 첨단소재를 제조하는데 필요한 고순도 은을 다양한 자원으로부터 회수하는 공정을 개발하는 것은 매우 중요하다. 본 논문에서는 여러 자원으로부터 은의 침출을 위해 개발된 공정을 조사하였다. 무기산(질산과 황산)과 무기산 및 산화제(오존, 산소, 과산화수소, 3가 철이온)의 혼합용액에 의한 은의 침출공정의 장단점을 비교하였다. 또한 thiourea와 thiosulfate에 의한 은의 침출과 무기산에 의한 침출에 대해 환경에 미치는 영향을 중심으로 비교하였다.

주제어 : 은, 습식제련, 침출, 2차 자원

Abstract

The special properties of silver are often indispensable in the manufacture of advanced materials. Therefore, it is of importance to develop a process to recover silver which is necessary for the production of advanced materials from diverse resources. In this manuscript, the developed processes for the leaching of silver from diverse resources are reviewed. For this purpose, the advantages and disadvantages of using some inorganic acids (nitric and sulfuric acid) and their mixture with other oxidizing agents (ozone, oxygen, hydrogen peroxide and ferric ion) were investigated. Moreover, the leaching of silver with thiourea and thiosulfate was compared over those by inorganic acids in terms of environmental effect.

Key words : Silver, Hydrometallurgy, Leaching, Secondary sources

1. Introduction

Silver is an important metal widely used in the manufacture of advanced materials due to its special physical

and chemical properties. Generally, silver is recovered from either some natural resources as by-product or the residues after the extraction of other metals¹⁾. In recent years, with the decrease in the amount of resources

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containing silver, the secondary resources become more important in the production of advanced materials containing silver. Along with technology innovation, more intense application of silver to advanced materials has been tried, such as nano-silver, solar cells, printed inks, battery catalysts, printed circuit boards and so on²⁻⁶. The consumption of silver is rapidly increasing in accordance with the demand for metallic silver from the industries. Therefore, development of a hydrometallurgical process to recover silver with high purity from secondary resources is of utmost importance.

There are lots of resources containing metallic silver, such as pyrrargyrite, Au-Ag ore, lead-zinc sulfide concentrates, refractory antimony ore, plumbojarosite, lead-silver concentrate, gold-antimony alloy, manganese silver ore etc. In recent years, pure silver is employed in the manufacture of the electronic products and other advanced materials. Therefore, it is very important to recycle the secondary resources, such as the printed circuit boards of waste mobile phones or computers, medical X-ray process effluents⁷⁻⁹, photographic films¹⁰, crushed Au-Ag Wastes, silver sulphide precipitates, metallic scraps, anode slime, and spent catalyst¹.

In order to recover pure silver from secondary resources, metallic silver should be dissolved. Therefore, the first step in developing a process for the recovery of silver is to find economical as well as environment-friendly leaching condition to dissolve metallic silver. In most of the developed processes for silver, either inorganic acids or the mixture of acids and oxidizing agents are employed. The leaching processes to dissolve metallic silver from the secondary resources are reviewed in this work.

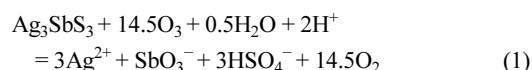
2. Developed Processes for the Leaching of Silver

2.1. Leaching with the mixture of inorganic acids and oxidants

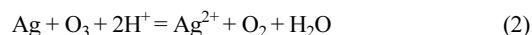
2.1.1. Leaching with the mixture of sulfuric and O₃

C. Rodríguez-Rodríguez et al.¹¹ developed aqueous ozone leaching of silver from the pyrrargyrite in acid solution and found that the concentration of ozone has

the greatest effect on the leaching percentage of silver. The solution containing 0.18 M sulfuric acid and 0.079 g O₃/L at 25°C is found to be suitable for leaching of silver. Under this condition, 80% of the silver was leached out from the pyrrargyrite within the reaction time of one hour. The overall reaction in this mixture can be represented as



J. Viñals et al.¹² used ozone to leach silver from the metallic scraps under the condition of 0.1 M H₂SO₄. According to this work, the concentration of hydrogen ion and ozone plays an important role during the reaction. Moreover, the main product was the insoluble Ag₂O when the pH ≥ 4 and the kinetics is controlled by mass transfer of ozone to the solid-liquid interface. Meanwhile, the main by-product O₂ could be converted by O₃ by Eq. (2), which could again dissolve metallic silver in the presence of acid.



2.1.2. Leaching with the mixture of (NH₄)₂S₂O₃ and NH₄OH

Chi Jung Oh et al.¹³ recovered precious metals from the print circuit boards of waste computers and the process is presented in Fig. 1. First, both Cu and Zn is removed by leaching with the mixture of H₂SO₄ and H₂O₂. Then the Au and Ag in the residue are dissolved by the mixture of (NH₄)₂S₂O₃ and NH₄OH. The leaching

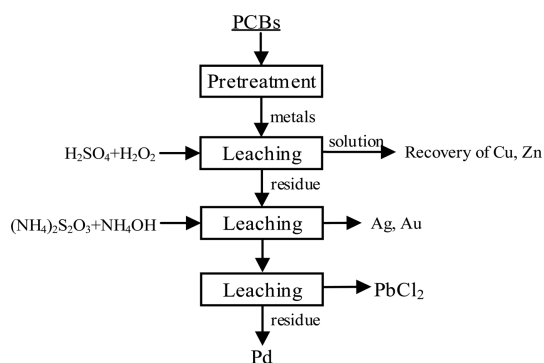


Fig. 1. Flowchart for the recovery of precious metals from PCBs.

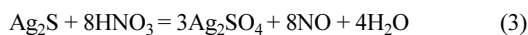
percentage of Au and Ag is 95% at 48 h and 100% at 24 h, respectively.

2.1.3. Leaching with HNO₃

Atefeh Khaleghi et al.¹⁴⁾ recovered silver and synthesized silver nanoparticles from the copper anode slime. All silver could be removed by 4 M nitric acid at the reaction temperature of 90°C at atmospheric pressure. At the same condition, only 26% leaching percentage of Ag was obtained by employing sulfuric acid. After leaching with nitric acid, Ag was separated from the leaching solution with HCl to synthesize silver nanoparticles with various methods.

Serdar Aktas¹⁵⁾ investigated the recovery of silver from spent silver oxide button cells by leaching with nitric acid. The leaching percentage of silver was 99% at 25°C, using 2.0 M nitric acid in 1 h. The dissolved silver was precipitated with KCl solution and reduced to metallic silver with zinc powder. N. Sathaiyan et al.¹⁶⁾ studied silver recovery from waste silver oxide button cells. In the case of leaching under the conditions (200 g/L HNO₃, 50°C), the leaching percentage of Ag was higher than 95%. And then pure metallic Ag was produced from the leaching solution.

P.C. Holloway et al.¹⁷⁾ leached the silver sulphide precipitates with nitric acid and the reaction is represented as Eq. (3). Under the condition of 150°C, 1100 kPa and the twice theoretical amount of nitric acid, the leaching percentage of Ag was 96%. From the leaching solution, silver with high purity was produced by hydrogen reduction at 150°C and 4000 kPa hydrogen pressure.



2.1.4. Leaching with nitrite-copper

G. Alvarado-Macías et al.¹⁸⁾ investigated the kinetics of silver leaching with the nitrite-copper system. Nitrite was employed as the oxidant as well as complexing agent for silver. It was found that the cupric-nitrite complexes have the synergistic effect on the silver leaching kinetics. The leaching percentage of Ag could be reached almost 92% under the conditions: 1.5 M nitrite; 0.2 M cupric ions; pH, 5; reaction time, 6 h. However,

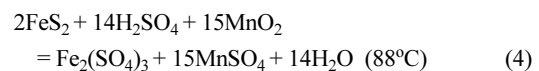
during the reaction, some copper precipitates were formed in the bulk solution.

Liu weifeng et al.¹⁹⁾ analyzed the behavior of silver and lead in the chlorination leaching process from gold-antimony alloy. When the chloride ion concentration was 2.4 M, the three quarters of silver complex species exist as AgCl₄³⁻. The reducing percentage of silver is 99% at the condition of 80°C and 1.5 h as the dosage of gold-antimony added is 10%.

R. Zárate-Gutiérrez et al.²⁰⁾ investigated the oxidative leaching of lead-silver concentrate. It was observed that 0.65 M nitric acid could dissolve 90% silver in 90 min. In addition, the presence of Fe³⁺ not only increased the solubility of silver, but also enhanced the kinetics. By the oxidation of Fe³⁺, most of the silver could be dissolved into solution at 130°C.

2.1.5. Leaching with cyanidation after pretreatment

Xian-yang Qiu et al.²¹⁾ treated a refractory Au-Ag ore with cyanidation after the pretreatment of reductive sulfating leaching as Eqs. (4) and (5). Through the pretreatment to overcome the hindrance by manganese, silver leaching was possible by employing the mixture of H₂SO₄, NaCN and the reductant of pyrite. The results indicated that the leaching percentage of silver sharply increased from 27 to 71% at the condition 0.2 g/L NaCN, pH 9, leach time 30 h, particle size 20 μm, FeS₂ 0.12 M. The disadvantage of this process is the high usage of the necessary chemicals. In addition, cyanidation has serious effect on the environment and health of worker.



Wei Li et al.²²⁾ reported the recovery of silver from manganese-silver ore followed by the pretreatment with ammonia-ammonium sulfate alkaline solution. When the conditions were 0.02 M NH₃·H₂O, 1.0 M (NH₄)₂SO₄, 4 g of Cu wire, a liquid- solid ratio of 3:1 and a stirring speed of 300 rpm, reaction time 4 h, the leaching percentage of Ag was 79%.

Ibrahim Alp et al.²³⁾ studied the extraction of silver

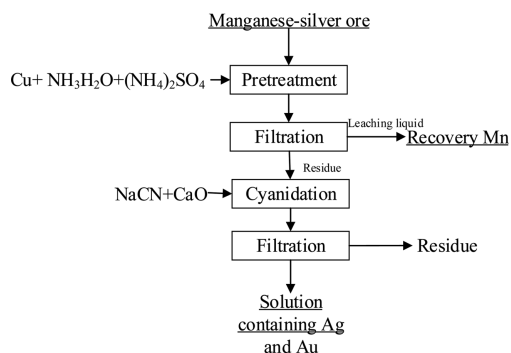


Fig. 2. The flow sheet of leaching Ag from manganese-silver ore.

and gold from a refractory ore with cyanidation followed by the KOH pretreatment. It was observed that under the KOH pretreatment the leaching percentage of silver and gold increased from 18.7 to 94.5% and 49.3 to 87.6%, respectively, at the conditions of 5 M KOH, 80°C, 2 h; cyanidation 1.5 g/L NaCN, pH 10.5, 24 h. The pretreatment could lessen the influence of other metals such as Sb on the leaching of Ag and Au. Oktay Celep et al.²⁴⁾ reported the effect of NaOH pretreatment on the leaching of silver and gold from a refractory antimonial ore. According to this work, the leaching percentage of silver and gold improved from 18.7 to 90% and 49.3 to 85.4% under the conditions: 3 M NaOH, 80°C.

2.1.6. Leaching with the mixture of HCl and CaCl₂

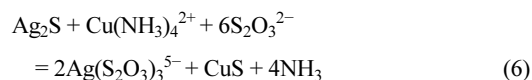
J. Viñals et al.²⁵⁾ reported the recovery of silver and gold from plumbojarosite by employing the system of HCl-CaCl₂. The leaching percentage of silver and gold was the same as 95% under the following conditions: CaCl₂, 300 g/L; temperature, 90°C; 900 mV versus SHE; pulp density 1: 2.5; reaction time, 1 h.

2.2. Leaching with thiosulfate

2.2.1. Leaching with thiosulfate

Bin Xu et al.²⁶⁾ reported the recovery of Ag, Au and Pd from de-copperized anode slime containing Sn, Pb and Sb with thiosulfate followed by sodium hydroxide and subsequent acetic acid pretreatment. It was found that Pb caused detrimental effect on the leaching of the precious metals. After the pretreatment for removing most

of Pb in the slime, the leaching percentage of Ag increased from 69.2 to 93.4%.



2.2.2. Leaching with ammonium thiosulfate

Jana Ficeriová et al.^{27,28)} used ammonium thiosulfate for silver recovery from sulfide concentrates. After mechanical activation and mechanochemical pretreatment, 99% Ag was dissolved into the solution at the conditions: reaction time, 15 min; stirring rates, 8.33 s⁻¹, temperature, 70°C; pH, 6; leaching reagents, 74 g/L (NH₄)₂S₂O₃ and 10 g/L CuSO₄·5H₂O. Jana Ficeriová et al.²⁹⁾ studied the leaching of Ag and Au with ammonium thiosulfate, after being pretreated by milling and found that 93% silver and 98% gold were achieved with 48 hours.

Katarzyna Wejman Gibas et al.³⁰⁾ recovered silver from a residue after pressure leaching of a flotation copper concentrate with thiosulfate. The optimum experimental conditions were 1 M thiosulfate, 0.8 M ammonia, 0.1 g/L, 25°C, 4 h. After the pressure NaOH leaching, the leaching percentage increased from 75 to 100%.

D.M. Puente-Siller et al.³¹⁾ developed a process for the leaching of silver with the system of thiosulfate-copper-NaOH-citrate. However, in the presence of EDTA and sodium citrate, the leaching process was affected due to the formation of a porous layer of copper sulfide and copper oxide.

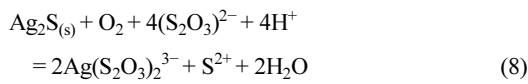
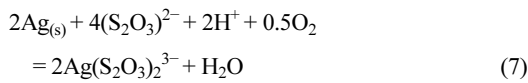
2.2.3. Leaching with the mixture of thiosulfate-nitrite-sulfite-copper

G. Alvarado-Macías et al.³²⁾ investigated silver leaching with the system of thiosulfate-nitrite-copper ions. It was found that the complexes of copper and ammonia can catalyze the silver dissolution. The sulfite ion has a negative effect on the Ag leaching, and at pH 9.6 the leaching of Ag was affected due to the formation of copper hydroxide species on the silver particle. The best conditions were: thiosulfate, 0.1 M; nitrite 1.5 M; copper, 0.05 M; pH 7.2. Under these conditions, the silver recovery was 96%. In 2016, G. Alvarado-Macías et al.³³⁾ found that thiosulfate concentration and temperature play

an important role on the leaching of silver in this system.

2.2.4. Leaching with Sodium thiosulfate and oxygen

Eleazar Salinas-Rodríguez et al.³⁴⁾ studied the kinetics of silver leaching from a mining tailing which contains metallic and silver sulfides with sodium thiosulfate. According to this work, the leaching reaction is controlled by mass transfer of oxygen at the solid-liquid surface and the reactions can be described as Eqs. (7) and (8).



I. Rivera et al.³⁵⁾ studied the dissolution kinetics of Ag in the thiosulfate solution in the presence of oxygen. The leaching process was controlled by mass transfer of oxygen in the solid- liquid interface and the stirring rate has an important influence on the transport of oxygen. In the presence of Cu and O₂, the leaching percentage increased by almost 30%. Xiong Tong et al.³⁶⁾ researched the role of oxygen and ammonium ions on the silver leaching in thiosulfate solution. It was found that ammonium and oxygen both can improve the dissolution of silver. However, the excess oxygen and ammonium will result in the degradation of thiosulfate.

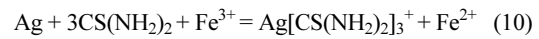
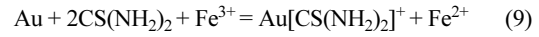
2.3. Thiourea leaching

2.3.1. Leaching with the mixture of thiourea and oxidants

Jana Ficeriová et al.³⁷⁾ utilized thiourea to recover Au and Ag from various Au-Ag wastes. In the thiourea solution, 98% of gold and 96% of silver were obtained in the presence of Fe³⁺ after 1 h. Particle size has an important effect on the dissolution of Au and Ag.

Li Jingying et al.³⁸⁾ studied the leaching of Au and Ag from the printed circuit boards of waste mobile phones with thiourea solution in the presence of Fe³⁺. Eqs. (9) and (10) represent the leaching reaction. At the conditions: particle size 100 mesh, 24 g/L thiourea, 0.6% Fe³⁺, after leaching reaction 2 h, the leaching percentage of Au and Ag was 90% and 50%, respectively.

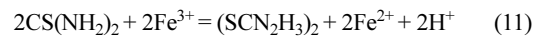
Since the leaching percentage of Ag is not high, it is necessary to do further research to improve the leaching percentage of silver.



2.3.2. Leaching with the mixture of thiourea and other treatment

Peter Baláz et al.³⁹⁾ reported the thiourea leaching of silver from a silver-bearing complex sulfide concentrate followed by mechanochemical alkaline leaching and the dissolution of silver increased from 5 to 90% in 10 min owing to the increase in the surface area of the concentrate from 0.3 to 15.7 m²/g by the pretreatment.

Ömer Yavuz et al.⁴⁰⁾ dissolved copper, silver and gold from anode slime by employing two steps. The first step was to dissolve 99% Cu and most of Se and Te with sulfuric acid in 1 h under 6 atm N₂ pressure at 350°C. The second step was to dissolve the residue after filtration with thiourea solutions in the presence of oxidants. When the Fe³⁺ concentration was 7.5 g/L, 97.8% thiourea was degraded and the degradation reactions of thiourea which occur during the reaction are represented as



2.3.3. Leaching with thiourea under the ultrasound

M. Salim Öncel⁴¹⁾ applied the ultrasound to the thiourea solution for silver leaching from solid waste of a cyanidation leach plant containing Ag₂Fe₃S₈, Ag₂S and Ag₃SbS₃. Under the conditions: particle size 55 μm, pulp density 100 g/L, thiourea concentration 7.4 g/L and H₂SO₄ concentration 28.7 g/L, reaction temperature 77°C, leaching time 24 min, the ultrasound power absorbed by the leaching solution was 80 W/l and the leaching percentage of silver reached 98.6%.

Jun Chang et al.⁴²⁾ made a comparison about the silver leaching between ultrasound and conventional methods from sintering dust in the acidic thiourea solution. The

experimental conditions were: particle size 75 ~ 96 μm , thiourea concentration 22 g/L, leaching temperature 50°C, stirring speed 300 rpm. Under these conditions, the leaching percentage of silver with conventional and ultrasound was 89.9% and 95%, respectively. The treatment by ultrasound could improve the diffusion through the product layer.

2.4. Leaching with the mixture of ionic liquid and aqueous acid

J.A. Whitehead et al.⁴³⁾ compared the leaching process of silver and gold from a sulfidic gold ore in the presence of oxidant (HSO_5^- or Fe^{3+}) and leaching agent (thiourea, chloride, bromide or iodide) in ionic liquids (1-alkyl-3-methyl-imidazolium). It was found that the system of ionic liquid have some advantages over aqueous system. In the same condition, thiourea was an efficient complexing agent. In addition, the leaching of silver and gold with thiourea in neutral bmimCl ionic liquid was affected by the oxidant and the HSO_4^- was better than Fe^{3+} .

3. Conclusion

This work reviewed the leaching process of silver from either natural or secondary resources. Considerable research has been done at a laboratory scale and the mixture of inorganic acids and oxidants, thiosulfate, thiourea and the mixture of ionic liquid with aqueous acid was investigated and discussed.

In the case of the inorganic acid such as nitric acid, the leaching percentage of silver was high but some gas was evolved during the leaching process. Thiourea and thiosulfate are non-toxic and non-corrosive reagents for dissolution of silver compared to cyanide leaching as well as other inorganic acids. Furthermore, thiourea and thiosulfate have higher recovery than other aqueous reagents, particularly the employment of oxidizing reagents such as oxygen, H_2O_2 and ferric etc. as well as some elements such as copper in the secondary resources could result in the higher consumption of these reagents. Therefore, further research should be performed to overcome these problems for the industrial application in future. In addition, the ionic liquid is an efficient solvent to

leach gold and silver from the mining with some leaching reagents, such as thiourea, chloride etc. It has some advantage compared to the aqueous system.

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