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Chemistry of Gold Extraction

Preface

Gold extraction or recovery from its ores may require a combination of comminution, mineral processing, hydrometallurgical, and pyrometallurgical processes to be performed on the ore. Gold mining from alluvium ores was once achieved by techniques associated with placer mining such as simple gold panning and sluicing, resulting in direct recovery of small gold nuggets and flakes. Placer mining techniques since the mid to late 20th century have generally only been the practice of artisan miners. Hydraulic mining was used widely in the Californian gold rush, and involved breaking down alluvial deposits with high-pressure jets of water. Hard rock ores have formed the basis of the majority of commercial gold recovery operations since the middle of the 20th century where open pit and or sub-surface mining techniques are used.

Editor

Contents

Chapter 1

Flame Atomic Absorption Determination of Gold Ion in Aqueous Samples after Preconcentration Using 9-Acridinylamine Functionalized γ-Alumina Nanoparticles

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ABSTRACT

A simple and sensitive solid phase extraction utilizing 9-acridinylamine functionalized alumina nanoparticles was developed, and their potential use for preconcentration and subsequent determination of gold by flame atomic absorption spectrometry (FAAS) was investigated. A number of parameters, namely, type, concentration, and volume of eluent, pH of the sample solution, flow rate of extraction, and volume of the sample, were evaluated. The effect of a variety of ions on preconcentration and recovery was also investigated. Gold ions were found to be recovered quantitatively at pH 3.0, with 0.1 mol L^{-1} thiourea in 2 mol L^{-1} H_2SO_4as eluent. The limit of detection (LOD), defined as five times the standard deviation of the blank, was determined to be lower than 13.0 ppb. Under optimum conditions, the accuracy and precision (RSD%) of the method were >98.0 and <1.5%, respectively. To gauge its ability in terms of application to real samples, the proposed method was successfully applied for determination of gold concentration in waste water samples and one soil standard material, and satisfactory results were obtained.

INTRODUCTION

The interest on gold is not only the reason of the use of gold in jewelry and its shining color, but also it has been found that gold can be used in catalytic converters, metallurgy, energy, electronics, health and environment, and many more applications [1, 2]. Nowadays, as human knowledge increases, more and more usage of gold has been investigated and it becomes more valuable. Waste water from mining, electroplating industries, electronics, and jewelry making manufacturing are the examples of natural samples which contain trace amount of gold [3, 4].

Several methods have been proposed for recovery of gold from waste water such as solvent extraction [5, 6], membrane disk [7], ion exchange [8, 9], coprecipitation [10, 11], cloud point extraction [12, 13], electrodeposition [14], leaching [15], chlorination [16], cyanidation [17], and solid phase extraction (SPE). Among all these methods, solid phase extraction has some advantages. It has low cost

and a high preconcentration factor, and also it is simple, rapid, and efficient [18].

The choice of adsorbent is an important stage in SPE due to control of the analytical parameters such as selectivity, affinity, and capacity [19]. So, different cartridges have been proposed including activated carbon [20], Dowex M 4195 [21], chitin [22], micro beads [23], thiol cotton fiber [24], chelating fiber [25], ion-imprinted polymers [26], modified resin [27, 28], polyurethane foam [29], activated alumina [30], and modified silica [31–34]. Among many types of adsorbents used in SPE, functionalized alumina has received great attention for their good mechanical and thermal stability and also less susceptibility to swelling or shrinking [35].

In this paper, for the first time, nanoparticles of alumina are functionalized with 9-acridinylamine group and used for extraction of trace amount of gold ions from some real samples of waste water. The optimum conditions including flow rates of the sample and eluent solution, pH of the solution, type, and least amount of eluent for elution were studied. Also, amount of break through volume, maximum adsorption capacity, and influence of various cationic interferences were investigated. This method can be applied as a reliable method for gold enrichment and determination in complex environmental samples.

EXPERIMENTAL

Reagents and Solutions

Analytical reagent grade chemicals—from Merck Company (Darmstadt, Germany) or Fluka Company (Buchs SG, Switzerland)—were employed for the preparation of all solutions. Gold standard solution of 1000 $\mu g\,mL^{-1}$ was purchased from Merck (Darmstadt, Germany). Deionized water was used during the experiments. The required pH adjustments were made by use of the buffer solutions. For the pHs 1 and 2, KCl/HCl buffer solutions were used. CH_3COOH/CH_3COONH_4 buffers were used to adjust pH in the range of 4–6, while NH_3/NH_4Cl buffers were used for pHs 8–10.

9-Acridinylamine, 3-(chloropropyl)-trimethoxysilane, CH_3COOH, $Na_3C_3H_5O(CO_2)_3$, Na_2HPO_4, NaH_2PO_4, HCl, and HNO_3 were purchased from the Merck, and thiourea and thioacetamide were purchased from Fluka.

Preparation of 9-Acridinylamine Functionalized Gamma Alumina Nanoparticles

Gamma alumina nanoparticles were prepared by the sol-gel method. Boehmite sol was prepared by the controlled hydrolysis of aluminum-tri-sec-butoxide. Details of the sol-gel synthesis are reported elsewhere [36]. The sol was dried at 50°C to obtain gel pieces and heat treated at 600°C to make gamma alumina. BET analysis shows 256 $m^2 gr^{-1}$ surface area for these nanoparticles. The average size of the individual γ-alumina nanoparticles was calculated as 93 nm using histogram program.

In order to synthesize 9-acridinylamine functionalized γ-alumina (py-γ-alumina), 1 g γ-alumina was activated in NaOH 2 M for 4 h then suspended in 50 mL toluene, and the mixture was stirred for 1 hour. After this step, 2.0 g of 3-chloropropyl trimethoxy silane was added and refluxed for 12 hours under nitrogen atmosphere. The white solid was removed from the solvent by filtration. The solid was suspended in 50 mL of triethylamine and toluene, and then 1 gr of 9-acridinylamine was added and refluxed for 6 hours. The white-brownish solid was removed from the solvent by filtration. After this step, it was washed by toluene and chloroform then dried at room temperature. Functionalization by pyridine was confirmed by IR spectroscopy and elemental analysis. IR spectroscopy is given as follows: IR (KBr, cm^{-1})—3445 (NH), 3023 (CH, aromatic) 2913 (CH, aliphatic), 1545 (C=C), 600–800 (alumina). Elemental analysis of py-γ-alumina sample gave 9-acridinylamine concentration of 0.49 mmol g^{-1}. The SEM photograph of py-γ-alumina nanoparticles is shown in Figure 1. A schematic diagram of synthesis route is shown in Figure 2.

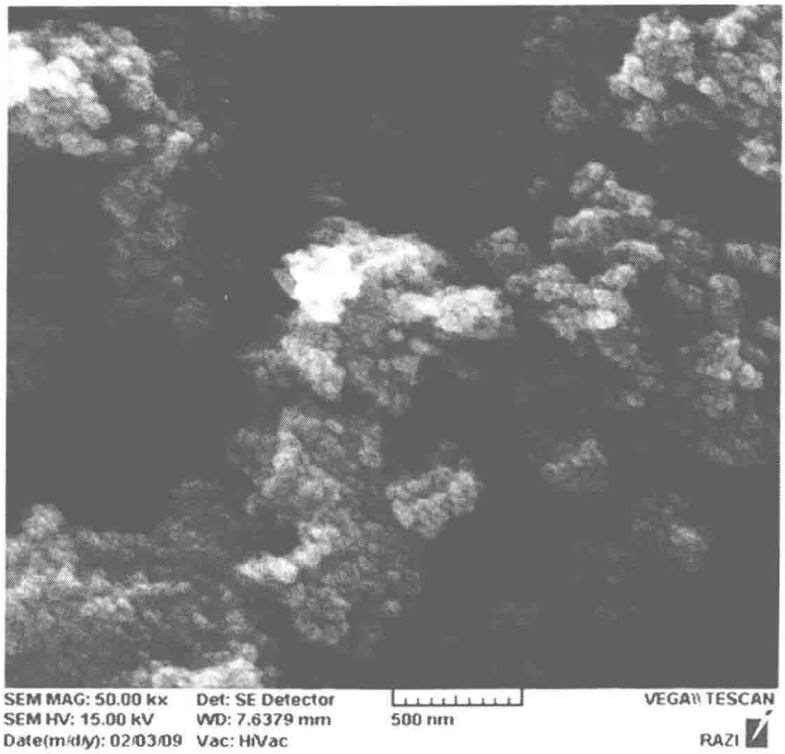

Figure 1: SEM photograph of 9-acridinylamine modified nano alumina.

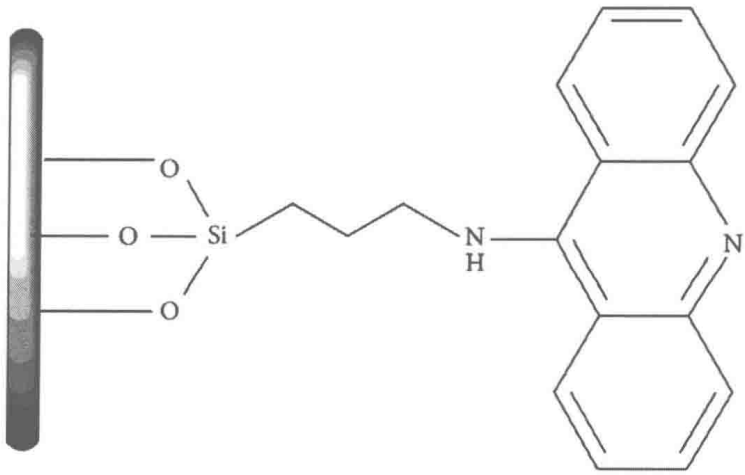

Figure 2: A schematic model for 9-acridinylamine modified nano alumina.

Instrument

A Shimadzu AA-680 atomic absorption spectrometer (AAS) equipped with single element hollow cathode lamp (6.0 mA for gold) and 10 cm of burner head and air acetylene burner was used for the determination of gold. The wave length at 242.8 nm (resonance line), the spectral band width at 0.5 nm, and the ratio of air-acetylene at 4.7 were set.

A digital WTW Metrohm 827 ion analyzer (Switzerland) equipped with a combined glass-calomel electrode at $25 \pm 1°C$ was used for the pH measurements.

A vacuum pump from Leybold (Germany) was used during the experiments, and an adjustable vacuum gauge and controller from Analytichem International (Harber City, CA) was used for adjusting flow rate during experiments.

Procedure

Column Procedure

A glass column with 120 mm in length and 2 cm in diameter was used for the experiments. It was filled with 200 mg of the alumina. Before using the column, it was washed with 5 mL dilute hydrochloric acid 1 M, 5 mL absolute ethanol, 5 mL toluene, and 20 mL distilled water to remove all organic and inorganic impurities.

Preconcentration Procedure

A solution containing 1 $\mu g\,mL^{-1}$ of gold was made. Solution's pH was adjusted to to 3 using $Na_3C_3H_5O(CO_2)_3$/HCl buffer solutions. Firstly, buffer solution was passed through the column to precondition it, then 100 mL of gold solution was passed at flow rate of 6 $mL\,min^{-1}$. The elution process was performed by passing 8 mL of thiourea 0.1 $mol\,L^{-1}$ in H_2SO_4 solution 2 $mol\,L^{-1}$. The eluted solutions were analyzed by FAAS for five times. The results were averaged and reported.

Sample Preparation

Real samples were obtained from tap water in Tehran, Caspian Sea, and jewelry waste water. The solutions were stored in cleaned polyethylene bottles and were filtered before usage. In order to validate the present method, a standard material sample (NCS DC 73323) with a certified gold content was obtained from China National Analysis Center for Iron and Steel. Standard material sample was digested with 6 mL HCl (37%) and 2 mL of HNO_3 (65%) in a microwave digestion system. The microwave program was as follows: 2 min at 250 W, 2 min without radiation, 6 min at 250 W, 5 min at 400 W, 8 min at 550 W, and then venting for 8 min. After digestion, it was diluted to 50.0 mL with deionized water. The pH of solutions was adjusted by adding $Na_3C_3H_5O(CO_2)_3$/HCl buffer solutions to 3. The proposed procedure was performed on the samples, and results were reported.

RESULT AND DISCUSSION

The effect of different parameters on gold extraction was studied. It was tried to find best conditions for extraction. The influence of pH, effect of type, concentration, and volume of eluent, and sample and eluent flow rates were studied, and the optimum values were obtained.

Influence of pH

Considering the important role of pH on solid phase extraction, the optimum condition for the pH was obtained by passing 100 mL of different sample solutions containing 1 mg L^{-1} gold ion with the pH range 2–9. Then, the column was washed with 8 mL of thiourea 0.1 mol L^{-1} in 2 mol L^{-1} H_2SO_4, and the eluent was analyzed with FAAS. The optimum condition for extraction was obtained at pH 3 (Figure 3).

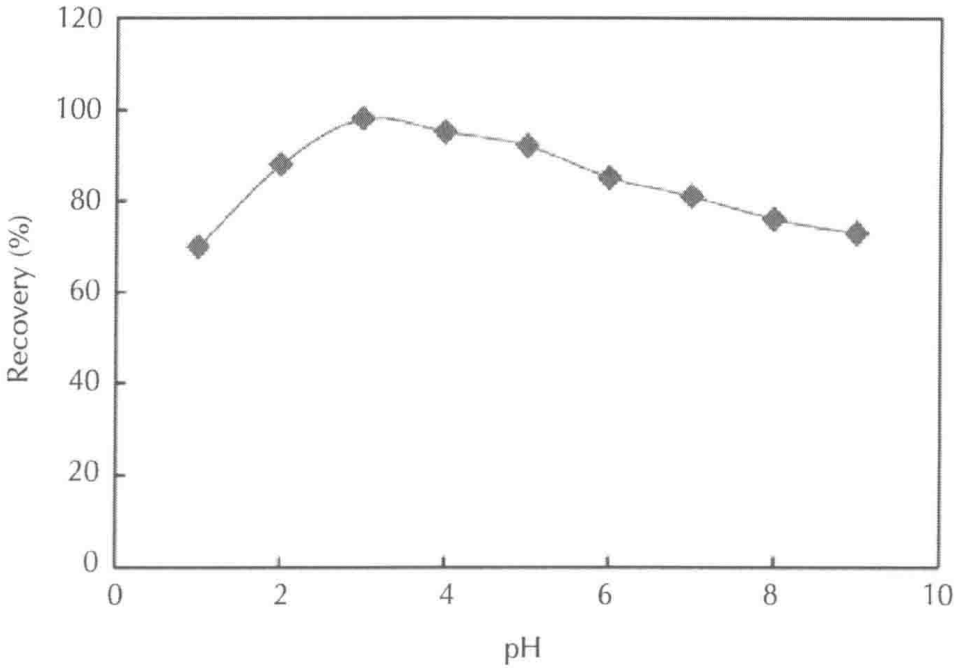

Figure 3: Effect of pH of sample solution on percent recovery of Au(III) by 9-acridinylamine alumina.

Effect of Type, Concentration, and Volume of Eluent

HCl, HNO_3, H_2SO_4, thiourea, and thioacetamide were mixed in different concentrations to make eluents. These eluents were testified to get the highest recovery in elution. It was observed that 0.1 mol L^{-1} thiourea in 2 mol L^{-1} H_2SO_4 solution provided effectiveness of the elution of Au (III) from sorbent. The optimum volume for elution was 8 mL of the eluent (Figure 4).