



ORIGINAL ARTICLE

Flame atomic absorption spectrometry determination of trace amount of gold after separation and preconcentration onto ion-exchange polyethylenimine coated on Al_2O_3



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Abstract The object of this work is to develop a simple and selective method for efficient extraction of Au(III) ions in aqueous solution using a new solid-phase extraction sorbent. Polyethylenimine (PEI) ion-exchange polymer was coated on alumina in the presence of $NaNO_3$. The method is based on sorption of Au^{3+} ions on 50 mg PEI/ Al_2O_3 . A solution of 0.5 M thiourea, then 1.0 M HCl effectively eluted the gold ion and then aspirated into flame atomic absorption spectroscopy (FAAS). The influence of flow rate of sample solution and eluent, the pH effect, eluent type and sorption capacity was investigated. The effects of various diverse ions for preconcentration and separation of the gold ion were investigated. Relative standard deviation of $4.0 \mu g mL^{-1}$ of gold was 1.46% ($n = 10$). The detection limit was $26.2 ng L^{-1}$ in original solution. The method has been applied successfully for the recovery of trace amount of Au(III) ions from water samples.

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1. Introduction

For many years, efforts of the analysts focused on the development of the extraction techniques that allow efficient extraction. There are traditional and modern extractions for separation, preconcentration and determination processes of trace metal ions from different matrices. When extracting liquid samples, traditional liquid–liquid extraction faces several limitations, namely use of an extractant non-miscible with the sample, difficulty in extracting polar and ionic compounds from water, large organic solvent volumes resulting in a diluted

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extract. To prevent these major drawbacks, solid-phase extraction has been employed (Camel, 2002). The preconcentration procedure is an essential step for the accurate measurement, especially those present in trace level ($\mu\text{g L}^{-1}$ and ng L^{-1}). The most common solid phase extraction (SPE) techniques for preconcentration of metal ions use various adsorbents such as, activated carbon (Ghaedi et al., 2007), cellulose (Burba and Willmer, 1983) amberlit XAD resins (Ferreria et al., 1999), analcime zeolites (Afzali et al., 2007), silica gel (Huang et al., 2008; Akhond et al. 2006), synthetic zeolites (Rios et al., 2008), octadecylsilica membrane disk (Bagheri et al., 2003) and other materials (Absalan et al., 2007; Absalan and Ayatollahi, 2003).

The ion-pair reagents containing bulky anions are often used to form extractable complex ion associates with charged bulky cationic complex species of neutral ligands (Aworn et al., 2005). Because of environmental and economical aspects, some studies were done on selective and effective extractants for toxic and valuable metals from water and soils. Au is known as the precious metal. This term reflects its economic value as well as the rare occurrences of the metals. Some papers have reported preconcentration and recovery of Au(III) in various samples with different kinds of methods (El-Shahawi et al., 2007; Adams, 2003; Zhang and Dreisinger, 2002; Donia et al., 2005; Chakrpani et al., 2001; Moreau and Bond, 2006; Chung and Tabata, 2002; Grosse et al., 2003).

The aim of this work is to develop a rapid and selective method for the extraction of gold(III) in aqueous solution, in level of ng L^{-1} , by using a, new sorbent, water-soluble polymer on alumina that form colored complexes with gold and are determined by flame atomic absorption spectroscopy (FAAS). This work improved the gold extraction techniques. The various parameters such as, pH, eluent solution, volume of sample, interfering ions and flow rates have been evaluated.

2. Experimental

2.1. Apparatus

A Varian model SpectraAA 220 atomic absorption spectrometer (Victoria, Australia, <http://www.varianinc.com>) was used for measuring of Au(III) in air-acetylene flame. The instrumental parameters according to manufacturer's recommendations are as follows; Wavelength, 242.8 nm; slit width, 1.0 nm; current lamp 4.0 mA; air flow 3.5 L min^{-1} and acetylene flow 1.5 L min^{-1} . Thermo Finnigan Flash EA1112 Microanalyzer was used for the determination of C.H.N percentage. A Metrohm 713 pH meter (<http://www.metrohm.de>) was used for pH measurement and equipped with a combined glass calomel electrode. Thermogravimetric analysis (TGA) was accomplished on a Perkin-Elmer TGA-USA.

Table 1 Elemental analysis (C.H.N.) for two kinds of sorbent.

Component name	PEI + alumina ^a	PEI + alumina + NaNO ₃ ^a
Nitrogen (%)	1.34594	1.47334
Carbon (%)	1.74820	1.95490
Hydrogen (%)	0.81426	0.84415

^a Average of two measurements.

2.2. Reagents

All reagents were of analytical grades. The stock solution ($1000.0 \text{ ng mL}^{-1}$) of Au(III) was prepared by dissolving appropriate amount of gold chloride (Bombay-New Dehli, Central Drug House) in HNO₃ 1:1 and dilute with distilled water to total volume of 50 mL. A 5% solution of polyethylenimine (PEI, molecular weight: 600,000–1,000,000) (Fluka) was prepared in distilled water. The sorbent was Al₂O₃ produced by Fluka. A solution 0.5 M thiourea (Merck) was prepared by dissolving thiourea in distilled water. Analytical grade salts of various metal ions (all from Merck) were of the highest purity available. Buffer solution with pH 5.7 was prepared from 0.1 M CH₃COOH and CH₃COONa.

2.3. Preparation of the sorbent

The coating PEI on alumina (giving PEI/Al₂O₃) was carried out as follows:

1.0 g alumina and 0.5 g NaNO₃, with 5 mL PEI 5% were mixed. Then pH was adjusted to 7.2. The obtained suspension was shaken for 24 h. The sorbent that was made, then filtered, rinsed with distilled water several times, and finally dried in desiccators. It was characterized with elemental analysis (C.H.N) and TGA. The thermogravimetric analysis curve of the PEI/alumina sorbent shows a four step mass loss up to 721 °C. The 1.7% mass loss up to 88.6 °C in the first step is due to sorbed water. In the second step mass loss is 3.9% up to 296.4 °C. In the third step mass loss is 3.9%, also, up to 506.5 °C and in the fourth step mass loss is 1.53% up to 723.1 °C. The mass losses in the second, third and fourth steps are corresponding with polyethylenimine. The results of elemental analysis of the dried sorbent showed that on new sorbent N and C elements have existed, thus PEI was coated on alumina (Table 1).

2.4. General procedure

The columns were packed with 50 mg of sorbent (PEI/Al₂O₃) and first was washed with an aliquot of water to wet the surface of the sorbent, then conditioned with a buffer solution at pH 5.7. Then, an aliquot of the sample solution containing of Au(III) (0.5–50.0 μg) was taken in a 50 mL beaker and 3 mL buffer solution was added with pH 5.7. The total volume of the solution was reached to about 30 mL with distilled water. This solution was passed through the column (funnel-tipped glass tube) with flow rate of 2 mL min^{-1} . With this procedure, Au³⁺ ions in water samples were quantitatively adsorbed on the new sorbent and consequently were eluted by 3.0 mL of 0.5 M thiourea and then 2.0 mL of 1.0 M HCl (final volume 5.0 mL) at a flow rate of 4 mL min^{-1} . The final solution was aspirated directly in to the flame AAS against the blank prepared in the same manner without the addition of gold.

3. Results and discussion

3.1. Sorption mechanisms

Polyethylenimine (PEI) is well known for its metal chelation property due to the presence of a large number of amine

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