

Research Article

Flame Atomic Absorption Spectrometric Determination of Trace Amounts of Silver after Solid-Phase Extraction with 2-Mercaptobenzothiazole Immobilized on Microcrystalline Naphthalene

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A simple and sensitive solid-phase extraction (SPE) procedure combined with flame atomic absorption spectrometry (FAAS) was designed for the extraction and determination of trace amounts of silver. A column of immobilized 2-mercaptobenzothiazole (MBT) on microcrystalline naphthalene was used as the sorbent. Silver was quantitatively retained on the column in the pH range of 0.5–6.0. After extraction, the solid mass consisting of silver complex and naphthalene was dissolved out of the column with 5.0 mL of dimethylformamide, and the analyte was determined by flame atomic absorption spectrometry (FAAS). Under the optimum experimental conditions, the adsorption capacity was found to be 1.18 mg of silver per gram of the sorbent. A sample volume of 800 mL resulted in a preconcentration factor of 160. The relative standard deviation obtained for ten replicate determinations at a concentration of $0.8 \mu\text{g L}^{-1}$ was 1.4%, and the limit of detection was $0.02 \mu\text{g L}^{-1}$. The method was successfully applied to the determination of silver in radiology film, waste water, and natural water samples. The accuracy was examined by recovery experiments, independent analysis by electrothermal atomic absorption spectrometry, and analysis of two certified reference materials.

1. Introduction

Silver and its compounds have an essential role in electronic and electrical applications, photographic film production, dental and pharmaceutical preparations, and manufacturing of fungicides [1, 2]. Silver is also used for the preparation of high-strength and corrosion-resistance alloys and jewelry. These widespread applications have resulted in an increased silver content of the environmental samples. Silver can also enter the environment via industrial waters because it is often an impurity in zinc, copper, antimony, and arsenic ores [3]. On the other hand, silver has been recognized as a toxic element to many aquatic organisms even at low concentrations [4]. Moreover, Ag accumulation in the human body may

lead to a permanent blue-gray skin discoloration (known as Argyria), and the threshold values regulating the maximum amount of silver permitted in different kinds of samples have been established in many countries [5]. Therefore, its trace determination in water and environmental samples is important.

Flame atomic absorption spectrometry is often accepted as a suitable instrumental technique for the measurement of silver because of its speed and ease of operation. However, it has the major drawback of low sensitivity for direct determination of silver in complex matrices [6]. Therefore, a separation/preconcentration step is often required prior to the ultra-trace determination of silver. The most widely used methods for the separation and preconcentration of the trace

amounts of silver ions are solid-phase extraction (SPE) [7, 8], solvent extraction [9], cloud-point extraction (CPE) [4], and dispersive liquid-liquid microextraction [10]. Among these methods, solid-phase extraction has gained rapid acceptance due to its simplicity, flexibility, high enrichment factor, and short analysis time. Appropriate organic ligands can be chemically or physically supported on various substrates, thus providing complexing or chelating sorbents for the separation and preconcentration of metal ions. Naphthalene is known as an excellent extractant at high temperature, but the procedure is time consuming and cannot be applied to thermally unstable complexes [11]. The extraction of metal chelates with microcrystalline naphthalene by adding an acetone solution of naphthalene is more rapid [12]. In recent years, column extraction of metal ions with modified microcrystalline naphthalene has attracted many researchers [13, 14].

2-Mercaptobenzothiazole (MBT) with sulfur and nitrogen donor atoms is known to form stable complexes with various metal ions. MBT is insoluble in water but soluble in many organic solvents. Thus, it can be applied to the separation and preconcentration of trace amounts of metals present in aqueous solutions. MBT has been used as the chelating agent for the preconcentration and spectrometric determination of Ag(I) [15–17], Cu(II) [18], Hg(II) [19, 20], Au(III), Pt(IV), and Pd(II) [21]. It has also been used in the construction of ion-selective and modified electrodes [22, 23].

In this study, a simple and efficient method for the selective separation and preconcentration of silver from a large volume of aqueous solution using a column of immobilized 2-mercaptobenzothiazole on microcrystalline naphthalene is described. The solid mass consisting of the silver complex and microcrystalline naphthalene is easily dissolved out of the column with a small volume of dimethylformamide (DMF), and the analyte is determined by flame atomic absorption spectrometry.

2. Experimental

2.1. Reagents. All of the chemicals were of the highest purity available from the Merck Company (Darmstadt, Germany) and were used without any further purification. Double distilled water was used throughout the experiments. A stock solution of 1000 mg L^{-1} of silver ion was prepared by dissolving an appropriate amount of AgNO_3 in water. Working solutions were prepared daily from the stock solution by appropriate dilution with distilled water.

2.2. Apparatus. An Analytikjena novAA 300 (model 330, Germany) atomic absorption spectrometer furnished with a silver hollow-cathode lamp and air-acetylene flame was used for all of the absorption measurements. The hollow-cathode lamp current was 4.0 mA, the wavelength was set at 328.1 nm, and the slit width was 1.2 nm. The pH measurements were carried out by a Metrohm pH meter (model 691, Switzerland) using a combined glass-calomel electrode.

2.3. Preparation of the Sorbent. MBT (0.4 g) and naphthalene (20 g) were dissolved in a hundred milliliters of acetone and then mixed with a magnetic stirrer at 35°C for 5 minutes. The mixture was then slowly added to 1000 mL of double distilled water at room temperature. The mixture was stirred for about 1 hour and was left aside for 120 minutes. Then, it was filtered through a sintered-glass funnel using the vacuum pump, and the residue was washed with distilled water for several times. Finally, the prepared sorbent was air dried and kept in a closed brown bottle for the subsequent use. The color of the prepared sorbent was yellow and it was stable at least for two months.

2.4. Procedure. The pH of an aliquot of the sample or standard solution containing 0.2–20.0 μg of silver was adjusted at ~ 1 with an appropriate amount of nitric acid. The solution was passed through a glass column packed with the MBT immobilized on the microcrystalline naphthalene (20 mm \times 10 mm i.d.) at a flow rate of 10.0 mL min^{-1} with the aid of a suction pump. The column was washed with a small amount of water, and the sorbent was pushed down with a flat glass rod to remove the excess of water attached to naphthalene. Finally, the solid mass consisting of the metal complex and naphthalene was dissolved with 5.0 mL of DMF, and the silver concentration in the resulting solution was determined by flame atomic absorption spectrometer.

2.5. Preparation of Water Samples. Water samples were filtered through $0.45 \mu\text{m}$ membrane Millipore filter. The pH was adjusted to ~ 1.0 with nitric-acid solution, and the analyte was determined according to the given procedure.

2.6. Preparation of Radiology Film. The proper amount of radiology film was washed with distilled water and dry ashed in a muffle furnace at 550°C for 60 min. The residue was treated with 10 mL of nitric-acid solution (6 mol L^{-1}) and evaporated to dryness at a low heating rate. The residue was dissolved in 80 mL of distilled water, filtered, and adjusted to pH 1.0 using nitric-acid solution. The clear solution was transferred into a 100 mL volumetric flask and diluted to mark with distilled water.

2.7. Certified Reference Materials. To proper amount of CPB-1 (Composition: Pb = $64.74 \pm 0.12\%$, S = $17.8 \pm 0.2\%$, Fe = $8.48 \pm 0.06\%$, Zn = $4.42 \pm 0.04\%$, SiO_2 = $0.74 \pm 0.04\%$, Sb = $0.36 \pm 0.03\%$, Al_2O_3 = $0.28 \pm 0.02\%$, Cu = $0.254 \pm 0.004\%$, As = $0.056 \pm 0.004\%$, Mn = $0.039 \pm 0.002\%$, Bi = $0.023 \pm 0.002\%$, Sn = $0.019 \pm 0.005\%$, Cd = $0.0143 \pm 0.002\%$, Ag = $626 \pm 6 \mu\text{g g}^{-1}$, Se = $30 \pm 3 \mu\text{g g}^{-1}$, and Hg = $5.5 \pm 0.5 \mu\text{g g}^{-1}$) or BCR No. 288 (Composition: Ag = $30.5 \pm 0.5 \mu\text{g g}^{-1}$, As = $55.7 \pm 1.6 \mu\text{g g}^{-1}$, Bi = $215.8 \pm 2.4 \mu\text{g g}^{-1}$, Cd = $33.3 \pm 0.9 \mu\text{g g}^{-1}$, Cu = $19.3 \pm 0.4 \mu\text{g g}^{-1}$, Ni = $4.57 \pm 0.11 \mu\text{g g}^{-1}$, Sb = $32.5 \pm 0.9 \mu\text{g g}^{-1}$, Se < $0.2 \mu\text{g g}^{-1}$, Sn = $30.6 \pm 1.5 \mu\text{g g}^{-1}$, Te = $32.8 \pm 1.3 \mu\text{g g}^{-1}$, Tl = $2.3 \pm 0.1 \mu\text{g g}^{-1}$, and Zn = $8.2 \pm 0.4 \mu\text{g g}^{-1}$), 5 mL of concentrated nitric acid was added and the solution was heated. Then, 3 mL of hydrogen peroxide was added, and the mixture was heated near to dryness.

The solution was diluted with distilled water and was filtered. The pH was adjusted to ~ 1 , and the solution was diluted to 100 mL in a conical flask.

3. Results and Discussion

The aim of this study was to develop a sensitive method for the separation and preconcentration of trace amounts of silver ions from an aqueous solution based on the selective adsorption of silver on a column packed with MBT immobilized on the microcrystalline naphthalene. MBT forms a strong water-insoluble complex with silver ions and permits the separation of silver at low pH. In order to obtain the best conditions for the extraction of silver, the procedure was optimized by the univariable method.

3.1. Influence of pH. The effect of the sample pH on the retention of silver was studied by varying the pH within the range of 0.5–9.0. The results showed (Figure 1) that the recovery of silver was maximized in the pH range of 0.5–6.0. The decrease in the extraction efficiency at $\text{pH} > 6.0$ is probably due to the precipitation of silver as its hydroxide. Thus, in order to achieve the maximum efficiency and selectivity of the silver extraction, a pH of ~ 1.0 was selected for the subsequent studies.

3.2. Choice of Solvent. The choice of an appropriate solvent for dissolving the Ag(I)-MBT along with naphthalene is an important factor. The solvent should completely dissolve the content of the column and should not interfere with the detection system; thus, with FAAS determination it must efficiently burn during the analysis of the sample. Various solvents were tested for dissolving the complex of Ag(I)-MBT immobilized on naphthalene. The solid material was insoluble in toluene, *n*-hexane, methyl isobutyl ketone, dioxane, and chloroform; however, it was readily dissolved in acetone, acetonitrile, and dimethylformamide (DMF). DMF was chosen because of its high capability in dissolving the sorbent, high stability, and compatibility with the FAAS. Furthermore, 5.0 mL of this solvent was found to be sufficient for complete dissolution of the solid mass.

3.3. Effect of Sample Flow Rate. Another important factor influencing the extraction efficiency and the speed of the analysis is the sample flow rate. In order to have a good precision, as well as sensitivity and speed, it is necessary to select a flow rate that guarantees the achievement of the equilibrium between the sample and the sorbent. The effect of the flow rate on the extraction recovery of silver was examined by varying the flow rate from 0.5 to 25.0 mL min^{-1} under constant experimental conditions. The outcome showed that (Figure 2) the extraction was relatively fast, and up to the sample flow rate of 12.0 mL min^{-1} the uptake of silver was constant and independent of the flow rate. Therefore, a sample flow rate of 10.0 mL min^{-1} was selected in further studies.

3.4. Effect of Sample Volume. In order to explore the possibility of enriching the trace amount of silver from the

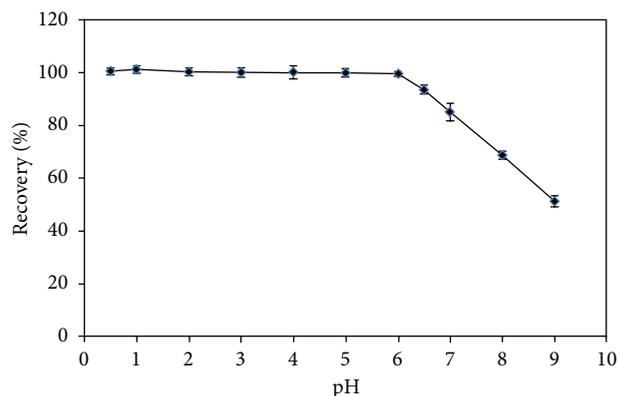


FIGURE 1: Effect of pH on the recovery of silver. Conditions: amount of silver 10 μg ; sample volume 50 mL; sample flow rate 5.0 mL min^{-1} ; solvent for dissolving adsorbent DMF (5.0 mL).

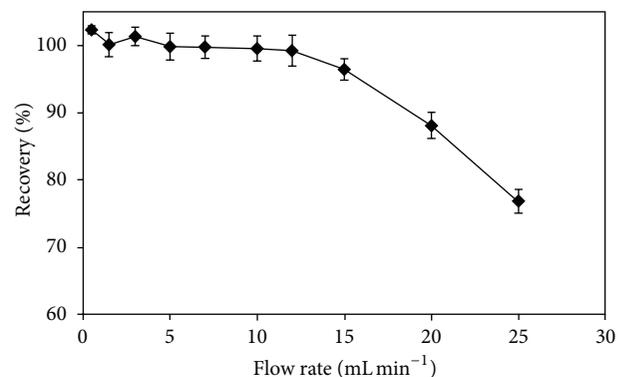


FIGURE 2: Effect of sample flow rate on the recovery of silver. Conditions: amount of silver 10 μg ; sample volume 50 mL; pH ~ 1.0 ; solvent for dissolving adsorbent DMF (5 mL).

large sample volume, different sample volume (50–1000 mL) containing 10 μg of silver was passed through the column. The retained analyte was then eluted with 5.0 mL of DMF, and the concentration of silver was determined. The results (Figure 3) showed that, up to an aqueous phase of 800 mL, the recovery was quantitative ($\geq 95\%$). Thus, the method has the capability of achieving a high preconcentration factor for silver ions.

3.5. Interferences Study. A possible concern was that whether the method can be employed for the determination of silver in real samples where other cations or anions might compete with the analyte and lower the extraction efficiency. For this reason, the effect of various ions on the recovery of 5 μg of silver from 100 mL of aqueous sample solution at an initial mole ratio of 1000 (ion/silver) was studied. When the interference was observed, the concentration of the interfering ion was lowered. A relative error of less than 5% was considered to be within the range of experimental error. The results of these studies (Table 1) indicated that the presence of high concentrations of the possible interfering ions in the sample had no significant effect on

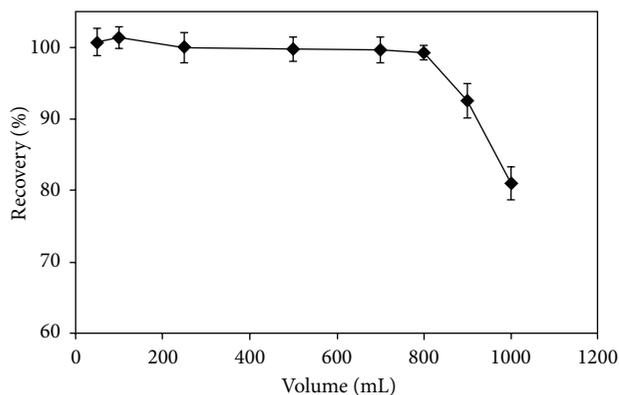


FIGURE 3: The influence of sample volume on the recovery of silver. Conditions: amount of silver $10 \mu\text{g}$; sample flow rate 10.0 mL min^{-1} ; pH ~ 1.0 ; solvent for dissolving adsorbent DMF (5.0 mL).

TABLE 1: Effect of foreign ions on the recovery of $5 \mu\text{g}$ of silver ions from 100 mL of aqueous solution.

Foreign ion	Mole ratio (M^{n+}/Ag^+)	Recovery (%)
K^+	1000	102.1 ± 3.1
Na^+	1000	99.9 ± 1.5
Mg^{2+}	1000	100.3 ± 1.4
Ca^{2+}	1000	99.7 ± 3.5
Ba^{2+}	1000	99.2 ± 1.9
Zn^{2+}	1000	99.4 ± 2.6
Co^{2+}	1000	102.8 ± 1.4
Ni^{2+}	1000	99.5 ± 1.0
Pb^{2+}	1000	98.1 ± 3.3
Cu^{2+}	1000	97.7 ± 2.1
NO_3^-	1000	97.6 ± 2.8
CH_3COO^-	1000	99.6 ± 3.0
SO_4^{2-}	500	99.1 ± 2.1
Cr^{3+}	500	99.0 ± 2.0
Cd^{2+}	500	99.3 ± 2.9
Fe^{3+}	500	104.3 ± 1.8
Cl^-	50	99.2 ± 1.0
Hg^{2+}	30	97.3 ± 1.0

the recovery of silver at trace levels. Furthermore, the effect of common ions present in the matrix of investigated samples such as Na^+ , K^+ , Ca^{2+} , Mg^{2+} , and Pb^{2+} at higher mole ratio (10000) was also considered, and no interference was observed. Thus, the method offers a high selectivity for silver ions.

3.6. Sorbent Capacity. The capacity of the sorbent for the retention of silver was determined. For this purpose, under the optimum pH, 1.0 g of the sorbent was added to the 100 mL of the solution containing $1500 \mu\text{g}$ of silver, and it was mixed for 30 minutes. The sorbent was then separated and the concentration of silver remaining in solution was determined by FAAS. The capacity of the sorbent for silver

was determined from the differences in the amount of the analyte in the initial and final solutions. The capacity of the sorbent for silver was found to be 1.18 mg g^{-1} of the sorbent.

3.7. Analytical Performance. Different concentrations of silver solution (800 mL) were processed according to the procedure, and it was found that the calibration graph exhibited linearity over the range of $0.15\text{--}25 \mu\text{g L}^{-1}$ of silver with a correlation coefficient of 0.9995. The equation of the calibration curve was $A = 0.0205C + 0.0009$ (where A is the absorbance and C is the concentration of silver in $\mu\text{g L}^{-1}$). The preconcentration factor defined as the ratio of the sample volume to the eluent volume was 160. The relative standard deviation (RSD) for ten replicate measurements of $0.8 \mu\text{g L}^{-1}$ of silver was 1.4%. The limit of detection defined as $3s_b/m$ (where s_b is the standard deviation of the blank and m is the slope of the calibration curve) was $0.02 \mu\text{g L}^{-1}$.

3.8. Application. The procedure was applied to the determination of silver ions in rain water, the Damavand spring water, well water, river water (taken from Karaj road, Karaj, Iran), waste water, and the radiology film sample. The reliability was checked by the recovery experiments, and the comparison of the results with the data was obtained by means of electrothermal atomic absorption spectrometry. The results of this investigation are indicated in Table 2. It can be seen that the recovery of the spiked sample is good, and at 95% confidence level there is no significant difference between the results of this study and the data obtained by electrothermal atomic absorption spectrometry. Furthermore, the proposed procedure was applied to the determination of silver in two certified reference materials, CPB-1 and BCR No. 288 with the silver concentration of $626.0 \pm 6.0 \mu\text{g g}^{-1}$ and $30.5 \pm 0.5 \mu\text{g g}^{-1}$, respectively. The concentrations of silver in CPB-1 and BCR No. 288 were found to be 618.0 ± 2.5 and $30.2 \pm 0.8 \mu\text{g g}^{-1}$, respectively, which are in good agreement with the accepted values. Thus, the method is suitable for the determination of silver in a wide range of samples.

3.9. Comparison of the Method with Other SPE Methods. The figures of merit of the proposed method and some other SPE methods combined with flame atomic absorption spectrometric for the determination of silver are summarized in Table 3. The proposed method in comparison with other methods showed some advantages such as a higher preconcentration factor and a lower detection limit.

4. Conclusion

The immobilized MBT on microcrystalline naphthalene is an effective sorbent for the separation and preconcentration of the trace amounts of silver ions from the aqueous solutions. The sorbent can selectively adsorb Ag(I) from the solution at low pH. So, the other heavy metals do not significantly interfere. The main advantages of the proposed method are

TABLE 2: Determination of silver in water samples and radiology film.

Samples	Added/ $\mu\text{g L}^{-1}$	Found ^a / $\mu\text{g L}^{-1}$	Recovery (%)	GFAAS ^a / $\mu\text{g L}^{-1}$
Well water	0	1.71 \pm 0.11	—	1.68 \pm 0.06
	10	11.50 \pm 0.36	97.9	—
Rain water	0	1.86 \pm 0.15	—	1.91 \pm 0.08
	10	11.78 \pm 0.23	99.2	—
River water	0	4.21 \pm 0.12	—	4.16 \pm 0.19
	10	14.15 \pm 0.18	99.4	—
Spring water	0	4.28 \pm 0.10	—	4.47 \pm 0.16
	10	14.23 \pm 0.27	99.5	—
Waste water	0	10.25 \pm 0.35	—	10.16 \pm 0.45
	10	20.06 \pm 0.54	98.1	—
Radiology film ^b	0	2.11 \pm 0.16	—	2.18 \pm 0.23
	1	3.07 \pm 0.35	96.0	—

^aMean and standard deviation of three independent determinations; ^bmg g⁻¹.

TABLE 3: A comparison between the figures of merit of the proposed method with similar SPE methods for Ag determination by FAAS.

Sorbent material	Reagent	pH	PF	DL/ $\mu\text{g L}^{-1}$	Reference
Polystyrene-divinylbenzene	6-Mercaptopurine	5.5–6.0	—	29	[24]
Silica gel	2,4,6-Trimorpholino-1,2,3-triazin	3.0–6.0	130	—	[25]
Silica gel	MBT	AM	—	0.66	[15]
Alumina	MBT	1.0–6.0	100	—	[16]
Alumina	Dithizone	1.0–9.0	100	—	[26]
Alumina	DDTC	1.0–7.0	125	0.7	[7]
Multiwalled carbon nanotubes	—	7.0–9.0	50	0.6	[27]
Moringa oleifera seeds	—	6.0–8.0	35	0.22	[28]
Naphthalene	MBT	0.5–6.0	160	0.02	This work

PF: preconcentration factor; DL: detection limit; Pb-DDTC: lead diethyl dithiocarbamate; MBT: 2-mercaptobenzothiazole; DDTC: diethyl dithiocarbamate; AM: acidic medium (0.05–6 mol L⁻¹ nitric acid).

the following: the ease of preparation of the sorbent, the high preconcentration factor (160), and the low detection limit (0.02 $\mu\text{g L}^{-1}$) which is suitable for the determination of silver in different real samples.

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