

Research Article

Determination of Trace Amount of Cadmium by Atomic Absorption Spectrometry in Table Salt after Solid Phase Preconcentration Using Octadecyl Silica Membrane Disk Modified by a New Derivative of Pyridine

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Silica-C18 bonded disk modified by a four-dentate Schiff base has been used for preconcentration of cadmium in table salt samples followed by flame atomic absorption spectrometry. The method is based on the adsorption of Cd on 1,2-bis(pyridin-2-ylmethylene) hydrazine as Schiff base ligand on silica-C18 disk. The effects of several factors such as type and concentration of the eluent, pH of sample solution, amount of ligand, and breakthrough volume have been optimized based on one variable at a time. In optimum conditions (type of eluent, HNO₃ 1.0 mol/L; volume of eluent, 5.0 mL; solution pH, 9.0; breakthrough volume, 1000.0 mL), preconcentration factor of the present method is about 200. The resultant limit of detection is 5.0 µg/L. Finally, the performance of the method has been evaluated for extraction and determination of Cd (II) in salt samples at milligram per liter concentration, and satisfactory results have been obtained (RSD ≤ 2.0%).

1. Introduction

The contamination of food with heavy metals could cause acute poisoning as well as long-term health problems. Table salt is an essential additive which is frequently added to certain foods for improving the taste, and it is used as a preservative as well. Therefore, due to the daily consumption of table salt, any contamination even in low level could put the consumer's health at risk. In fact, heavy metal ions are toxic, non-biodegradable, and tend to be accumulated in the human vital organs, where they can act progressively over a long period through food chains [1, 2], and the reported primary adverse health effects of them are lung cancer and kidney damage [3, 4]. Among these heavy metals,

cadmium is one of the most toxic elements for human and animals, even at low concentrations, and the International Agency for Research on Cancer classified Cd as a human carcinogen [5]. The FAO/WHO (Food and Agriculture Organization/World Health Organization) and Joint Expert Committee on Food Activities recommended maximum tolerable daily intake of cadmium from all sources (food, air, and water) of 1.0–1.2 µg/g weight of body [6]. In Codex standard the permissible level of cadmium in table salt is 0.2 µg/g.

Because of the complexity of sample matrix and low concentrations of cadmium in the environmental samples, preconcentration and matrix-removal steps are required to guarantee the accuracy and precision of the analytical

results [1, 5, 7]. Many preconcentration methods followed by different quantitative instrumental techniques have been investigated in trace heavy metal in the environmental samples [8].

Although atomic absorption spectroscopy (AAS) and inductively coupled plasma atomic emission spectroscopy (ICP-AES) are the most widely used methods, their sensitivity is usually insufficient for monitoring the low level concentration of these ions in the environmental samples [9–12]. Therefore, element with lower concentrations than the detection limits of the instrumental techniques are a serious problem in the trace heavy metal determination in the environmental samples [13]. The most common techniques reported for preconcentration of Cd (II) are liquid-liquid extraction (LLE) [14, 15], solid-phase extraction (SPE) [16, 17], ion exchange [18], cloud point extraction [19, 20], and electroanalytical techniques [21].

Solid-phase extraction (SPE) has advantages over other preconcentration methods in terms of simplicity, economy, rapidity, reusability of the adsorbent, ease of automation, higher preconcentration factor, lower consumption of reagents, and, more importantly, its environmental friendliness (use of less organic solvents). The most extensively used SPE sorbents are modified C-18 silica [22], activated carbon [23], alumina [24], and Amberlite XAD resins [25, 26]. For example, silica and silica-bonded adsorbents offer good advantages in terms of thermal, mechanical, and chemical stability. In addition, they act selectively towards a particular metal ion. In fact, the modified silica gel surfaces are known to act as a weak cation exchanger via its weak silanol groups, through immobilization of organic complexing agents, either chemically or physically. These modified surfaces greatly enhance metal exchange capacity and improve selectivity of these phases to metal ion removal, separation, or preconcentration prior to their determination by X-ray fluorescence or AAS analysis [11, 27–30]. Chelating agents can be easily loaded on silica-bonded adsorbents with good stability and Schiff bases often used as chelating ligands and their metal complexes have been of great interest for many years. Chelating ligands containing N, S, and O donor atoms are of special interest because of the various ways in which they are bonded to the metal ions. The variety of possible Schiff base metal complexes with wide choices of ligands and coordination environment has encouraged us to choose a tetradentate Schiff base for modifying the adsorbent disk. The mobilization and immobilization of cadmium have been shown to depend significantly on the complexation of the metal center by chelating nitrogen donor ligands [8, 31–33].

In the present method, octadecyl silica disk modified with a new derivative of pyridine ligand (tetra-dentate Schiff base), (Figure 1) which is followed by flame atomic absorption spectrometry, has been applied for preconcentration and determination of cadmium in salt samples because of the toxic environmental impact of Cd. The effects of different variables such as type and concentration of eluent, sample solution pH, concentration of ligand, and effect of breakthrough volume have been investigated and optimized based on the method of one variable at a time. In the end, the

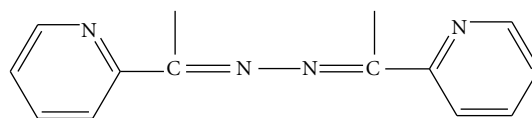


FIGURE 1: Structure of (1,2-bis(pyridin-2-ylmethylene) hydrazine) ligand.

optimized method has been applied for determination of Cd ions in real samples.

2. Experimental

2.1. Chemicals and Reagents. Acetonitrile (ACN), methanol, nitric acid, ethylene diamine tetraacetic acid (EDTA), acetic acid, sulfuric acid, hydrochloric acid, phosphoric acid, sodium dihydrogen phosphate, ammonium acetate, ammonia solution, and ammonium chloride salt are of analytical grade from Merck (Darmstadt, Germany). Double distilled water has been prepared by Mili-Q system from Millipore (Bedford, MA, USA).

Reference standard solution of cadmium having the metal concentration of 1000.0 ± 2.0 mg/L in 2% (v/v) of HNO_3 have been purchased from assurance company. The working standard solutions have been prepared by appropriate dilution of the stock solution with double distilled water, all solutions have been stored in ambient temperature. The new synthesized Schiff base (Figure 1) with highest purity has been used as the chelating ligand.

Reference standard solutions of As, Hg, Pb, Na, K, Ca, and Mg (each of them 1000.0 ± 2.0 mg/L in 2% (v/v) of HNO_3) have been bought from Merck company.

For preparing pH 2.0 and 3.0 buffer solutions, appropriate amount of phosphoric acid has been added to sodium dihydrogen phosphate solution (0.1 mol/L). Moreover, appropriate amount of acetic acid has been added to ammonium acetate solution (0.1 mol/L) to give buffer solutions of pH 4.0–6.0. Titrisol buffer solution from Merck ($\text{C}(\text{KH}_2\text{PO}_4) = 0.026$ mol/L and $\text{C}(\text{Na}_2\text{HPO}_4) = 0.041$ mol/L) has been used as pH 7.0, and an ammonium ammonia buffer solution has been prepared by adding an appropriate amount of ammonia to ammonium chloride solution (0.1 mol/L) to give a solution of pH 8.0–10.0.

2.2. Instrumentation. A Varian model AA-1275 flame atomic absorption spectrometer with a deuterium background corrector was used for determination of Cd ions, and a cadmium hollow cathode lamp was used as light source operated at 3.5 mA. The wavelength was set at 228.8 nm resonance line and the spectral band pass at 0.5 nm, and the measurements were carried out in an air/acetylene flame. A digital pH meter, Mettler Toledo model MP225, equipped with a combined glass calomel electrode was used for the pH adjustments.

Extraction was performed with 47 mm diameter \times 0.5 mm thickness Empore membrane disks containing octadecyl-bonded silica ($8 \mu\text{m}$ particle, 60-Å pore size, 3 M Co., St. Paul, MN) with a standard Millipore 47 mm filtration

apparatus. The twin cylinder vacuum pump model of TC-2000 V (Taiwan) was used to provide the required pressure for extraction.

2.3. Extraction Procedure. The membrane disk was conditioned with 20.0 mL of methanol and 20.0 mL of water, respectively, to remove all contaminants arising from the manufacturing process and the environment. The disk was dried by passing the air through it for several minutes, and then 8 mg of ligand was dissolved in 3.0 mL ACN and passed through sorbent several times under gravity until the ligand penetrated the membrane completely. Then modified disk was applied for extraction step, 100.0 mL of standard solution containing 10.0 μg of Cd was prepared, and solution pH was adjusted with buffer solution at 9.0. Afterwards, this solution was loaded at a flow rate of about 4.0 mL/min with the aid of a vacuum pump, and the retained Cd ions were eluted with 5.0 mL of HNO_3 (1.0 mol/L) under gravity (about 2.0 mL/min) followed by FAAS for determination of Cd content. Five measurements were made for each sample and the results were subsequently averaged. After each extraction, the disk eluted with two times 5.0 mL of HNO_3 (1.0 mol/L) and with 5.0 mL of water, respectively.

2.4. Sample Preparation. One salt sample from mine (Rahrahak mine, Iran) and a commercial sample from Iranian market were collected as the real samples. 2.0 gr table salt was dissolved in 60.0 mL double distilled water, and solution pH was adjusted to 9.0 with 5.0 mL of ammonium chloride/ammonia buffer solution and diluted to 100.0 mL with water. This solution was applied for the preconcentration step.

3. Results and Discussion

In the proposed method, solid phase extraction with silica-C18 disk, modified by a four-dentate Schiff base, which is followed by FAAS determination was applied for preconcentration of Cd ions in salt samples. An azine compound (1,2-bis(pyridin-2-ylmethylene)hydrazine), (Figure 1), was used as a selective chelating ligand for cadmium complexation. The effect of different parameters on the extraction and elution of Cd ions was optimized by using the method of one variable at a time.

3.1. Effect of Solution pH. Among chemical variables, pH is seemed to be the most important parameter for adsorption of the metal ions on ligand. As Schiff bases are conjugate bases of weak acid groups, they have very strong affinity for hydrogen ions. The pH of solution will determine the values of conditional stability constants of metal complexes on the surface of the sorbent. In order to assess the effect of solution pH on extraction efficiency, the pH of 100.0 mL model solutions containing 0.1 mg/L of Cd ions was adjusted in the range of 3.0–10.0 with different buffer solutions. The retained Cd ions were eluted with 5.0 mL of HNO_3 (1.0 mol/L) followed by FAAS for determination of Cd content. Figure 2 reveals that extraction recovery of the analyte grows significantly by

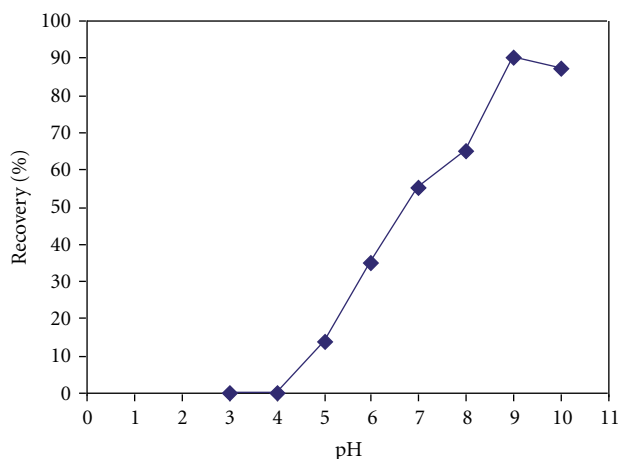


FIGURE 2: Effect of solution pH on recovery (%) (extraction conditions: sample volume, 100.0 mL; eluent solvent, 5.0 mL of HNO_3 (1.0 mol/L); amount of ligand, 8 mg; concentration of Cd ion, 0.10 mg/L).

increasing the pH of sample solution, but it is important to know that the pH higher than 10.0 may cause the destruction of the disk. Therefore, pH = 9.0 was selected as the best pH in the subsequent experiments.

3.2. Effect of Type and Concentration of Eluent. Complete stripping of Cd ions from ligand was examined with several eluents such as H_2SO_4 , HNO_3 , HCl, and EDTA. In fact, the selection of the eluent was really difficult due to the limitation of FAAS to tolerate organic solvents, and the eluent should not destroy the solid phase. In lower pH, the ligand–Cd complex dissociates to release Cd ions. Therefore, acidic eluents are the best choice to obtain efficient extraction. According to the results in Figure 3, HNO_3 was selected as the best eluent. HNO_3 at various concentrations was investigated for increasing recovery. The results show that HNO_3 with 1.0 mol/L concentration is the best solvent for elution of Cd ions.

3.3. Effect of Ligand Concentration. Prior to adsorption of trace heavy metal ions on a solid phase for preconcentration, metal ions were converted to a suitable form including metal chelates or metal inorganic complexes. Because of this, different concentrations of ligand were investigated for increasing extraction efficiency. Different amounts (3, 5, 8, and 10 mg) of ligand in 3.0 mL of ACN were added to the disk, and quantitative recoveries were obtained for cadmium ions with 8 mg of ligand. In all further works, 8 mg of ligand in 3.0 mL ACN solution was applied for modification of the disk.

3.4. Stability of Solid Phase. The reuse of the modified disk was tested for its ability to perform the SPE for more than one sample. It was found that the use of the same disk modified with Schiff base for at least twenty times did not change the recovery of Cd ion. In addition, there was no decrease

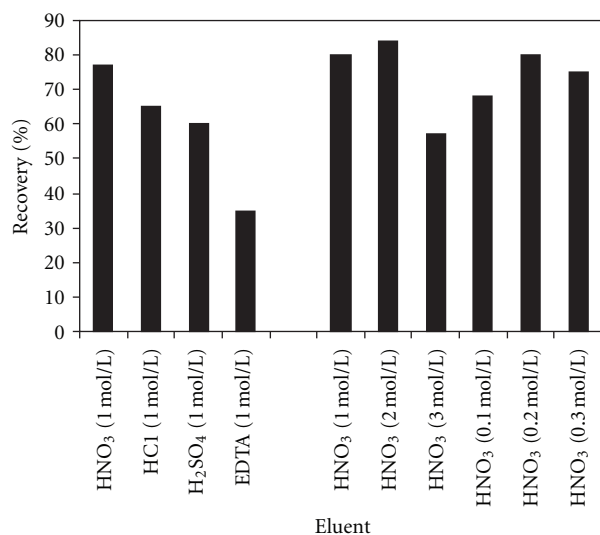


FIGURE 3: Effect of type and concentration of eluent on recovery (%), (Extraction conditions: sample volume, 100.0 mL; amount of ligand, 8 mg; sample solution pH, 9.0; concentration of Cd ion, 0.10 mg L⁻¹).

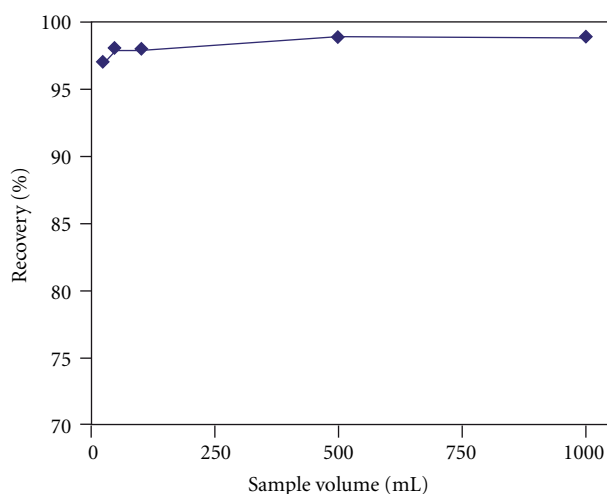


FIGURE 4: Effect of breakthrough volume on recovery (%) (extraction conditions: eluent solvent, 5.0 mL of HNO₃ (1.0 mol/L); amount of ligand, 8 mg; sample solution pH, 9.0; concentration of Cd ion, 0.10 mg L⁻¹).

in recovery for interday and intraday extractions. However, for the SPE of trace amounts of Cd from real samples, it is recommended to use a new modified disk for each sample to protect them from possible contaminations.

3.5. Effect of the Breakthrough Volume. The breakthrough volume of sample solution was tested by treating standard solutions containing 10 μg Cd ions with different sample volumes (25.0, 50.0, 100.0, 250.0, 500.0, and 1000.0 mL). The effect of breakthrough volume on recovery has been showed in Figure 4. In all cases, the extraction by membrane disk was found to be quantitative. Thus, 1000.0 mL was the maximum sample volume in which quantitative extraction of Cd ions is

TABLE 1: Extraction of Cd ion from binary mixtures.

Interference ion	Concentration (mg/L)	Recovery of Cd (II) (%)
Mg ⁺²	300	96.0 \pm 0.5
Ca ⁺²	1500	95.0 \pm 1.0
K ⁺	100	94.0 \pm 1.0
Hg ⁺²	100	94.0 \pm 1.0
As ⁺³	100	95.0 \pm 0.5
Pb ⁺²	100	89.0 \pm 1.0

possible. Sample volume more than 1000.0 mL was not tested due to increase of extraction time. To reduce the extraction time, 100.0 mL sample solution was selected.

3.6. Sorption Capacity. The maximum capacity of the membrane disk modified by a new Schiff base ligand was determined by passing 50.0 mL of an aqueous solution containing 500.0 μg Cd ions through the disk. The amount of the retained Cd ions determined using furnace atomic absorption spectrometry. The maximum capacity of the disk was found to be 325 μg Cd ion per 8 mg of ligand.

3.7. Influence of Interference Ions. The impact of a variety of cations found in salt samples on the determination of Cd has been studied. The limit of heavy metals (Pb, Cd, As, Hg, Cu, Fe) and other cations (K⁺, Mg²⁺, Ca²⁺) is determined in Codex standard for table salt. Therefore, the above-mentioned cations have been chosen for the investigation of interference ions. For this purpose, Na⁺, K⁺, Mg²⁺, Ca²⁺, As²⁺, Pb²⁺, and Hg²⁺ with limited concentrations in accordance with Codex standard mentioned in Table 1 were added individually to 100.0 mL aliquots of solutions containing 10 μg of Cd. The same procedure for extraction on solid phase by Schiff base was followed. The effect of Cu and Fe ions was not investigated because of precipitate formation in the working pH.

3.8. Analytical Performance. Regression equation, correlation coefficient (R^2), limit of detection (LOD), and preconcentrations factor (PF) were calculated in optimal conditions and summarized in Table 2.

The LOD was calculated as the analyte concentration equal to three times the standard deviation of the blank signal divided by the slope of the calibration curve. The LOD was obtained in the range of 5.0 $\mu\text{g/L}$.

The PF was calculated as the ratio of sample solution volume to eluent solution volume. The PF obtained for solution containing 100.0 $\mu\text{g/L}$ of Cd (II) was 200.

The real samples were extracted by C18-bonded silica disk modified with the tetradentate Schiff base followed by FAAS. Analysis of samples showed that they contained trace amount of Cd (II) which were lower than permissible limit in Codex standard. Therefore, all the real salt samples were spiked with reference standard solution at 100.0 $\mu\text{g/L}$ concentration to assess the matrix effects.

TABLE 2: The limits of detection, regression equations, correlation coefficient, preconcentration factor, recovery, and relative standard deviation for extraction method.

Analyte	Regression equation	R^2	LOD ($\mu\text{g/L}$)	PF	Recovery (%)	RSD (%)
Cd ion	$Y = 0.368X + 0.126$	0.9905	5.0	200	98.0 ± 0.5	≤ 2

TABLE 3: Determination of Cd ions in the salt samples.

Sample	C_{added} ($\mu\text{g L}^{-1}$)	C_{found} ($\mu\text{g L}^{-1}$)	Recovery (%)	RSD (%)
Rahrahak sample	100.0	93.7	93.7	1.7
Commercial sample	100.0	95.4	95.4	1.4

Table 3 shows that the results of the four replicate analyses of each sample obtained by the SPE/FAAS method are satisfactorily in agreement with the amounts of Cd (II) added. It is noticeable that the amount of Cd ion was not detectable in all samples without spiking.

4. Conclusion

This paper describes a preconcentration/separation procedure based on solid phase extraction of cadmium in salt samples using an octadecyl silica disk modified by a new pyridine derivative ligand followed by flame atomic absorption spectrometric determination. The proposed method has advantages such as high sample cleanup, efficiency, selectivity, and preconcentration factor. In conclusion, the performance of this procedure has been excellent in extraction of trace amounts of Cd (II) from different salt samples. Furthermore, this method is very environmental friendly, and it can be used in complex matrices with high selectivity.

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