



Application of L-cystine modified zeolite for preconcentration and determination of ultra-trace levels of cadmium by flame atomic absorption spectrometry



Seyyed Ahmad Rezvani^a, Ahmad Soleymanpour^{a,b,*}

^a School of Chemistry, Damghan University, Damghan 3671641167, Iran

^b Institute of Biological Science, Damghan University, Damghan 3671641167, Iran

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ABSTRACT

A very convenient, sensitive and precise solid phase extraction (SPE) system was developed for enrichment and determination of ultra-trace of cadmium ion in water and plant samples. This method was based on the retention of cadmium(II) ions by L-cystine adsorbed in Y-zeolite and carry out in a packed mini-column. The retained cadmium ions then were eluted and determined by flame atomic absorption spectrometry. The scanning electron microscopy (SEM), powder X-ray diffraction (XRD) and Fourier Transform Infrared (FT-IR) spectroscopy techniques were applied for the characterization of cystine modified zeolite (CMZ). Some experimental conditions affecting the analytical performance such as pH, eluent type, concentration of sample, eluent flow rate and also the presence of interfering ions were investigated. The calibration graph was linear within the range of 0.1–7.5 ng mL⁻¹ and limit of detection was obtained 0.04 ng mL⁻¹ with the preconcentration factor of 400. The relative standard deviation (RSD) was obtained 1.4%, indicating the excellent reproducibility of this method. The proposed method was successfully applied for the extraction and determination of cadmium(II) ion in black tea, cigarette's tobacco and also various water samples.

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1. Introduction

Application of an effective extraction and preconcentration step in analysis of environmental samples with ultra-trace levels of pollutant results in the rise of analyte concentration and reduction of matrix interference [1]. Solid phase extraction (SPE) is a technique widely used for preconcentration of analytes in diverse samples. SPE possesses several advantages, including large preconcentration factors, high recovery, rapid phase separation, low cost, low consumption of organic solvents, elimination of matrix interference and the ability of combination with different detection techniques in the form of on-line or off-line mode [2,3]. The choice of sorbent is the most critical parameter in optimization of the SPE extraction because it can control the analytical parameters such as selectivity, affinity and capacity of the extraction [4]. The sorbent used in SPE should have a stable and insoluble porous matrix with suitable functional groups to interact with analytes and its sorption

must be fast and quantitative. Moreover, it should have high sorption capacity, good regenerability and surface area accessibility [5]. A variety of solid materials such as modified or functionalized activated carbon [6–10], fullerenes [11], carbon nanotubes [12,13], nanocomposite materials [14], magnetic nanoparticles [15–17], thiol cotton [18], resins [19–21], bonded or modified silica [22–24], sisal fiber and cellulose [25,26], polyurethane [27], imprinted polymers [28–30] and modified bentonites [31,32] have been successfully used for the separation and preconcentration of trace amounts of heavy metal ions from different environmental samples.

Zeolites are synthesized in various environments such as hydrothermal environments, sedimentary rocks, alkaline igneous rocks, miscellaneous joints and cavities. They are porous hydrated aluminosilicates where the SiO₄ tetrahedral units form three-dimensional supercages. Depend on the shape of the pores, the nanopores are often called cages or channels and usually filled with water as well as charge-balancing cations [33]. Substitution of some Si atoms by Al atoms results in a negatively charged structure that arises from the excess negative charge of (AlO₄)⁵⁻ relate to (SiO₄)⁴⁻ [34]. These negative sites are balanced by exchangeable alkaline and alkaline earth metal cations (e.g., Na⁺, K⁺, Ca²⁺ and Mg²⁺).

* Corresponding author at: School of Chemistry, Damghan University, Damghan 3671641167, Iran. Fax: +98 23 35220095.

E-mail address: soleymanpour@du.ac.ir (A. Soleymanpour).

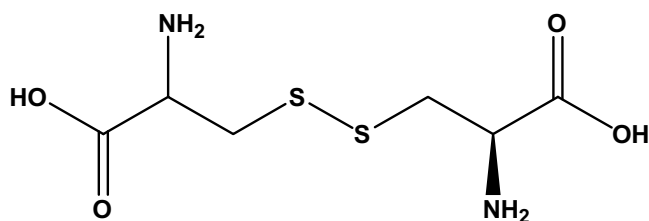


Fig. 1. Molecular structure of cystine.

Thus, zeolites can have ion exchanger properties and so they have been used as ion exchangers for water purification, drying agents or absorbents for organic vapors, molecular sieves, separating membranes, catalysts and detergent builders in industry, agriculture, veterinary medicine, health care and environmental protection [35,36]. The adsorption capacity and especially ion-exchange properties of zeolites have been investigated in analytical separation and preconcentration of various species. Therefore, different physical and chemical methods have been developed to modify zeolites in order to improve the extraction performance, such as adsorption capacity, selectivity and sensitivity to primary analytes [34,37].

Amino acids are among the most interesting potential metal-binders with high affinity to a wide range of metals because they can coordinate through their oxygen, nitrogen and sometimes sulfur atoms [38]. Selectivity, strong binding capacity and environmental innocuity are some properties that make amino acids ideal molecules for modification of sorbents to preconcentrate metal ions [39]. Cystine (Fig. 1), the oxidized form of cysteine, is a good complexing agent and absorbent for metal ions due to the presence of two carboxyl groups, two amino groups and two sulfur atoms in its structure [40].

Cadmium is one of the most toxic heavy metals which has been known to damage kidney, liver, lungs, bones and blood cells when people expose to it at levels above the maximum contaminant level (MCL) for long-term [41]. This toxicity could be due to a direct or an indirect interaction taking place between the metal ion and the biological system such as enzyme and other ligands in cells like hydroxyl, carboxyl, cysteinyl and histidyl side chains of proteins [42]. The American Environmental Protection Agency (US EPA) set the cadmium MCL at $10 \mu\text{g L}^{-1}$ for the standard drinking water [43]. Various applications of cadmium in different fields such as paint industries, alloy processing, electric batteries, semiconductors, plastics, besides natural exposure of cadmium from earth crust and soils, may result in cadmium uptake in waters and soils, and as a consequence in food stuff [44,45]. Therefore, determination of trace levels of cadmium in environmental samples has great importance.

The aim of this work is to develop a new rapid, high sensitive and efficient method for selective extraction and preconcentration of trace amounts of Cd(II) ions from aqueous media using Y-zeolite modified with L-cystine as a new sorbent. The significance of this work is the possibility of using a cheap and simple modified adsorbent for the preconcentration of trace amounts of cadmium ions. The analytical performance of the sorbent was investigated and the procedure was successfully applied for the determination of cadmium in different water and plant samples.

2. Experimental

2.1. Apparatus

The cadmium analysis was performed with a Spectra CTA-2000 flame atomic absorption spectrometer (FAAS) (Chem Tech Analytical) and inductively coupled plasma optical emission spectrometer (ICP-OES) (GBC, Integra-XL). The cadmium hollow cathode lamp was run under manufacturer recommended conditions of 4.0 mA

current, 228.8 nm wavelength, and a bandwidth of 1 nm. All measurements were carried out in an air/acetylene flame. The rate of acetylene and air flow was set up at 2.2 L min^{-1} and 7.2 L min^{-1} , respectively. All pH measurements were performed with a Metrohm 870 model digital pH meter. A funnel-tipped glass tube ($100 \times 5 \text{ mm i.d.}$) was used as a column for extraction and preconcentration. All glassware and columns were washed with nitric acid (2 mol L^{-1}) and double distilled water before application. FT-IR spectra were obtained as KBr pellets on a Perkin-Elmer spectrum RXI FT-IR spectrophotometer. Morphology of sorbent was observed on a ZEISS Sigma VP scanning electron microscope (SEM). The X-ray diffraction (XRD) analysis was performed using a D8 Advance Bruker with $\text{CuK}\alpha$ ($\lambda = 0.15406 \text{ nm}$) radiation over the range of $4\text{--}70^\circ$ two theta.

2.2. Reagents and solutions

All of the chemical used were of the highest purity available and used without any further purification except for vacuum drying. Doubly distilled water was used throughout for preparing all aqueous solutions. The Y-zeolite used in this study was obtained from Negin Powder Co., Semnan, Iran. The stock solution of cadmium(II) ($100 \mu\text{g mL}^{-1}$) was prepared by dissolving appropriate amounts of cadmium nitrate (Merck, Germany) in 0.1 mol L^{-1} nitric acid. Standard solutions of cadmium(II) were prepared daily by dilution of cadmium ion stock solution. A solution of cystine ($1.0 \times 10^{-3} \text{ mol L}^{-1}$, Merck) was prepared in pH 7.4. Sodium acetate, acetic acid and sodium hydroxide were purchased from Merck and used in preparation of acetate buffer. The pH of buffer solution was adjusted at 5.5 by the addition of sodium hydroxide to a solution containing sodium acetate and acetic acid with a total concentration of 0.02 mol L^{-1} . Nitric acid solutions (0.2 , 0.5 , 1.0 and 2.0 mol L^{-1}) and hydrochloric acid solutions (2.0 mol L^{-1}) were prepared by dilution of concentrated solutions (Merck). The solution of ethylenediaminetetraacetic acid (EDTA) was prepared by dissolving of the appropriate amount of EDTA- $2\text{H}_2\text{O}$ in water.

2.3. Modification, sorption and extraction procedure

Y-zeolite (300 mg) was loaded into a glass column which plugged with a certain amount of glass wool at the ends. A certain amount of glass wool was also placed on the top of sorbent to avoid disturbance during the test. The column was first washed with water and then 5.0 mL cystine solution ($2.5 \times 10^{-3} \text{ mol L}^{-1}$) was passed through the column by the flow rate of 0.25 mL min^{-1} . Then, the column was washed with 2.0 mL water in order to remove the weakly adsorbed cystine. The FT-IR spectrum showed adsorption of an appropriate amount of cystine into the zeolite. A 100 mL of cadmium solution in acetate buffer was passed through the column at the optimum flow rate. After sorption step, adsorbed cadmium ions were eluted with 2.0 mL of 2.0 mol L^{-1} nitric acid with appropriate flow rate. The analyte concentration in the eluted solution was finally determined by FAAS.

2.4. Optimization of experimental factors

In order to access the best extraction performance some affecting experimental factors were optimized. Thus, the influence of pH on the recovery of cadmium(II) ions was investigated in the pH range of 2.0–10.0 by adjusting the pH of working solutions with the addition of hydrochloric acid or sodium hydroxide solutions. To examine the effect of sample flow rates on the adsorption of Cd(II) ions, 100 mL solution containing $0.5 \mu\text{g}$ cadmium(II) were passed through the column at various flow rates (controlled by a peristaltic pump). The effect of sample volume was investigated by 25–1000 mL volumes of the analyte solutions containing $0.5 \mu\text{g}$

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