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Complete List of Authors:	Mohajer, Samira; Ferdowsi University of Mashhad, chamsaz, mahmood; Ferdowsi University of Mashhad, Chemistry; ferdowsi university of mashhad, chemistry Entezari, Mohaamad Hassan; Ferdowsi University of Mashhad, Chemistry

Ultratrace determination of cadmium(II) ions in water samples using graphite furnace atomic absorption spectrometry after separation and preconcentration by magnetic activated carbon nanocomposites

Samira Mohajer*, Mahmoud Chamsaz, Mohammad H. Entezari

Department of Chemistry, Faculty of Sciences, Ferdowsi University of Mashhad, Mashhad, Iran

ABSTRACT

The main purpose of the present study is sorption/preconcentration of trace amounts of cadmium using magnetic activated carbon nanocomposite (MAC) as an adsorbent in various water samples using graphite furnace atomic absorption spectrometry (GFAAS). The advantages of this method include the elimination of organic solvent and easily separation of magnetic nanocomposites from the bulk of solution by applying a magnet without any preliminary centrifugation and filtration steps. Initially, MAC was prepared by sonochemical approach and characterized by FT-IR and TEM. Cadmium ions from 100 mL sample solution was then adsorbed on the prepared sorbent by batch mode at pH 7. After sedimentation of MAC, quantitative desorption occurs using 0.5 mL of 0.5 mol L^{-1} HCl. The effects of various parameters on solid-phase extraction procedure efficiency including pH, sample volume, equilibrium time, type and concentration of eluent have been thoroughly investigated and optimized. Under the optimal experimental conditions, detection limit (LOD) based on three times of the standard deviation of the blank signals (n=8) was 4 ng L^{-1}. The relative standard deviation (RSD) resulting from the analysis of five replicate solutions containing 100 ng L^{-1} Cd(II) was 3.5%. The effect of potentially interfering ions on the percent recovery of cadmium ions was also studied. The accuracy of the method was verified using the analysis of a certified reference material. The proposed method was successfully applied to determine Cd(II) ions in aqueous real solutions.

***Keywords**:* Cadmium(II), Water samples, Graphite furnace atomic absorption spectrometry (GFAAS), Magnetic activated carbon nanocomposite

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* Corresponding author. Tel.: +98 511 8795162; fax: +98 511 8796416.

E-mail address: mohajersamira@yahoo.com

1. Introduction

Determination of ultratrace amounts of heavy metals due to their impacts in environmental water pollution is of great importance. Analysis of cadmium as a heavy metal is highly demanded because of its high level of toxicity and wide distribution.[1] Cadmium is widely used in various industries such as alloys, batteries, metal pleating and also as stabilizer and pigments in plastics.[2,3] Entering cadmium to surface water, as a result of industrial activities, has increased the probability of human exposure to this element. Due to high toxicity of cadmium, even at low concentrations, its acceptable level in drinking water by World Health Organization (WHO) has been declared to be 3 μg L^{-1}.[4] Cadmium accumulation in the body, due to its high biological half-life, causes numerous damages to organs such as the lungs, liver and kidney.[5,6] Therefore, determination of trace amounts of cadmium in environmental water samples by employing a sensitive, selective and reliable analytical method is of great importance.

Many methods mainly spectrophotometry,[7-9] flame atomic adsorption spectrometry (FAAS),[10-13] electrothermal atomic adsorption spectrometry (ETASS)[14,15] and inductively coupled plasma optical emission spectrometry (ICP-OES)[16-18] have been used for determination of cadmium in different samples. However, low concentration of cadmium in real samples, as well as high matrix effects has hindered its analysis. In this respect, some pretreatment steps for separation and preconcentration of cadmium to lower detection limit and enhance the sensitivity of detection techniques are inevitable.[19] These include liquid liquid extraction (LLE)[20,21] and solid phase extraction (SPE).[22-25] SPE is superior to LLE for complex matrices, due to its simplicity and speed, ease of automation, improved sensitivity, high enrichment factor and low consumption of organic solvents.[26]. Recently, solid phase extraction have been widely used for environmental sample pretreatment in order to separate/enrich cadmium ions.[27-33]

The most commonly used adsorbent in SPE is activated carbon (AC) for its large surface area, high adsorption capacity and porous structure.[34,35] Nowadays, biomass is considered superior for production of activated carbon because it is inexpensive and renewable and represents a greener technology compared to fossil fuels.[36-38] The AC regeneration from sample solution due to carbon fouling and generation of secondary waste has limited its applications in many fields. To overcome these drawbacks, magnetic composite was prepared by combination of activated carbon with magnetic particles.[39] Synthesized magnetic composites could be rapidly separated from the bulk of solution using an external magnetic field.

Now days, special attention has been made towards magnetic nanocomposites as newly synthesized nanoadsorbents due to their large surface area, highly active surface sites and ease of separation.[40,41] Magneic nanocomposites are superparamagnetic and hence the adsorbed analytes on their surface can be quickly removed from the matrix by the use of a magnet. They have been extensively employed for dyes and organic compounds removal from aqueous solutions.[42-44]

To the best of our knowledge, no work has been reported dealing with the application of magnetic activated carbon nanocomposites for separation and preconcentration of metal ions so far. This paper reports the synthesis of magnetic activated carbon nanocomposites (MAC) from carrot dross and evaluates the feasibility of employing this nanocomposite for preconcentration of Cd(II) ions in water samples for the first time. The structure of this sorbent were confirmed using transmission electron microscopy (TEM) and Fourier transform infrared (FT-IR). The main factors influencing the adsorption process such as pH, sample volume, contact time, etc. were thoroughly investigated and optimized

2. Experimental

2.1. Instrumentation

Cadmium ions was determined by Graphite Furnace atomic absorption spectrometry (GF-AAS) using a Shimadzu model AA-670 apparatus. Deuterium background correction was employed to correct the nonspecific absorbance. The heating program applied for cadmium determination is given in Table 1. The synthesis of nanocomposite was carried out with Sonics and Material, 750 watt, 20 kHz. Ultrasonic irradiation was performed in a water-jacketed rosette-cell with 6 cm internal diameter and 11 cm height. The 20 kHz wave was emitted by titanium probe (diameter 1.1 cm) and power supply of VCX 750 W, with piezoelectric lead zirconate titanate crystal (PZT). The pH values was adjusted using a Metrohm Model 632 pH meter supplied with a glass-combined electrode. An ultrasonic bath (Branson 1510, Danbury, CT) was used to disperse the nanoparticles into the solution. A Nd-Fe-B magnet (10.0 cm × 5.0 cm × 2.0 cm) was used for magnetic separation.

2.2. Standard solutions and reagents

Analytical grade reagents were purchased from Merck unless otherwise specified and deionized water was used throughout (18.2 MΩ cm^{-1}). All glasswares were soaked in diluted nitric acid for more than 12 h and washed with deionized water before use. A stock solution of 1000 mg L^{-1} Cd(II) was prepared by dissolving an appropriate amounts of $Cd(NO_3)_2$ in deionized water with the addition of nitric acid. Working solutions of the cadmium were prepared freshly by suitable dilutions of the stock solution with deionized water. Standard stock solutions (1000 mg L^{-1}) of other metal ions were prepared from their salts.

2.3. preparation of magnetic activated carbon

The magnetic activated carbons was prepared by Entezari et.al[45] as follows: definite amounts of iron(II) chloride and iron(III) chloride with a mole ratio of 1/2 with respect to metal ions was dissolved in 50 mL of milli-Q water and transferred into a 100 mL beaker followed by adding 4 mL of ammonia solution (25%). The solution was vigorously stirred until the pH of the solution has reached 9 and the black magnetite sol was obtained. The magnetite sol was then poured into a rosette-cell followed by addition of 7 g AC powder and the mixture was sonicated for 30 minutes at 40 °C. Finally, the solid phase containing MAC was separated by a magnet, washed several times with milli-Q water and then dried at room temperature.

2.4. Extraction and preconcentration procedure

Batch adsorption studies were performed as follows: 100 mL of aqueous sample solution was transferred into a glass beaker. 30 mg of MAC was added to the solution and pH of the mixture was adjusted at 7 by drop-wise addition of diluted NaOH solution. The adsorbent was dispersed into the sample solution by ultrasonication for 3 min in order to facilitate the adsorption of Cd(II) ions. The MAC was then isolated from the suspension under an external field via a piece of permanent magnet. The adsorbed cadmium(II) ions on nanoparticles were eluted by 0.5 mL of 0.5 mol L^{-1} HCl and analyzed for its cadmium content using GFAAS.

2.5 Sample preparation

Tap water from our research laboratory (Ferdowsi University, Mashhad, Iran), sea water (Caspian Sea), spring water (Dehsorkh, Neyshaboor, Iran) and mineral water from local store were collected analyzed after pH adjustment.

A standard reference material, JR-1 (Igneous rocks), was analyzed to validate the accuracy of the method. 0.2 g of this material was weighted accurately in a Teflon beaker and dissolved in a mixture of 7 mL HF, 0.7 mL HNO_3 and 2.3 mL H_2SO_4. The resulting solution was heated on a hot plate at low temperature for 12 h until completely decomposed and evaporated nearly to 2 mL. 8 mL concentrated HNO_3 was then added to the solution together with heating until a clear solution obtained and it was diluted to 25 mL with deionized water.

3. Results and discussion

The optimization study for preconcentration process was carried out in order to achieve maximal recovery for an efficient extraction of ultratrace amounts of Cd(II) ions.

Characterization of the adsorbent

The synthesized MAC was characterized by TEM and FT-IR. Fig. 1 shows TEM images of MAC nanocomposites. As can be seen, the nanocomposite particles are very fine and diameter of the particles is estimated to be less than 10 nm. The FT-IR spectrum of Fe_3O_4, AC and MAC are shown in Fig. 2. The AC spectrum shows three distinct peaks at 3440 cm^{-1},1620 cm^{-1} and 1120 cm^{-1} which could be assigned to OH, C=O and C-O vibrations, respectively. In the case of MAC, the peaks at 3440 cm^{-1} and 1583 cm^{-1} belong to OH and C=O vibrations, respectively. The broad peak at 1123 cm^{-1} is suggested to be as the result of coating of magnetic nanoparticles on the surface of AC. The FT-IR spectrum of Fe_3O_4 nanoparticles shows a peak at 550 cm^{-1} which is due to the M-O band. Therefore, the small peak at 590 cm^{-1} in MAC spectrum could be attributed to the Fe_3O_4 nanoparticles coated on the surface of AC.

Effect of pH

The pH of the aqueous solution is an important controlling parameter for improving the adsorption efficiency and reduction of interferences from the matrix because pH affects the distribution of active sites on the surface of sorbent.[46] The effect of pH on the extraction recovery of cadmium ions was studied in the pH range of 1-10 according to the recommended procedure. The results show that the adsorption efficiency increased by increasing the pH up to 5.0 (Fig. 3) and leveled off at higher pHs. Lower recoveries at pH values below 5 can be explained by competing H^+ with Cd^{+2} ions in the solution for the active sites of sorbent.[2,47] Hydroxide ions at higher pHs than 10 would complex and precipitate Cd(II) ions, and therefore the separation may not be due to adsorption phenomena.[48,49]

Zeta potential measurement at different pHs was also used for justification of the above results. The pH value at which the Zeta potential equals zero is called the isoelectric point and it is used for qualitative assessment of the absorbent surface charge. The IEP of MAC was found to be around 4.5. At pHs lower than 4.5, the adsorbent has positive surface charge and consequently the interactions between the adsorption sites on nanocomposite and cadmium ions are electrostatically repulsive. On the other hand, the cadmium adsorption capacity will increase at pHs above the IEP due to columbic attraction which can readily take place between cadmium ions and negatively charged surface of the nanoadsorbent.

To ensure the highest recovery of adsorption, pH of 7 was employed as the optimum value.

Effect of eluent

Because the adsorption of cadmium was low at pHs lower than 4.5, HCl at different concentrations was tested for desorption of the Cd(II) ions from the adsorbent surface. The results indicate that, 0.5 mol L^{-1} HCl provided higher recovery for elution of Cd(II) ions. The volume of HCl for quantitative elution of Cd(II) ions was also examined and quantitative recoveries (>90%) was obtained with 0.5 mL of 0.5 mol L^{-1} HCl.

Effect of sample volume

In order to improve the preconcentration factor, a large volume of sample solution is required. For this purpose, different volumes of sample solutions in the range of 25-200 mL was examined by applying the general procedure (Section 2.4). As shown in Fig. 4, quantitative recoveries (>90%) was obtained with sample volumes up to 100 mL. Therefore, sample volume of 100 mL was selected as the optimum value for subsequent experiments. Using elution volume of 0.5 mL, a 200-fold preconcentration factor was achieved by this method.

Effect of sorbent amount

Nanoparticles have high surface area and therefore low amounts of nanosorbent can achieve satisfactory results than conventional sorbents for quantitative adsorption of ions. In order to investigate the influence of MAC amounts on the recovery of cadmium, the extraction was carried out by varying the amounts of sorbent in the range of 1 to 50 mg. The results are shown in Fig. 5. It can be seen that 30 mg of sorbent was adequate for quantitative retaining of the Cd(II) ions and so this amount was chosen for further experiments.

Effect of ultrasonication time

In the SPE process, the contact time is an important factor in order to realize complete extraction. Therefore, the effect of ultrasonication time was investigated in the range of 1-15 min time intervals for both adsorption and desorption stages. The results show that quantitative recoveries for both processes was achieved when ultrasonic time was 3 min due to high surface area to volume ratio of MAC nanocomposites. Hence, 3 min was selected as the optimum time for subsequent experiments.

Effect of sedimentation time

Due to the superparamagnetism properties of MAC, adsorbent could be completely separated from the bulk of solution using an external magnetic field in a very short time. No significant benefit was observed for quantitative recovery of Cd(II) ions when the sedimentation time was greater than 1 min. Therefore, 1 min sedimentation time was taken as the optimum value for subsequent experiments.

Adsorption capacity

One of the important parameters for assessment of adsorbent characteristics is adsorption capacity, which is defined as the amount of adsorbent required for quantitative recovery of analyte from the solution. According to Maquieira et al[50] criteria, the profile of adsorption isotherm is assessed by plotting the concentration of analyte versus the amount of sorbed analyte per gram of nanoparticle (Fig. 6). As can be seen, the maximum value of adsorption capacity for MAC was found to be 93.8 mg g^{-1} for cadmium. A comparison between adsorption capacity of synthesized MAC nanocomposite in this work with some adsorbents in literature for extraction of cadmium is summarized in Table 2. It is clear that the proposed magnetic nanocomposite has adsorption capacity superior to some of the reported adsorbents.

Regeneration of sorbent

In order to check the stability and applicability of synthesized nanoparticles at the experimental conditions, they were subjected to several cycles of extraction and back extraction. The results show that, 30 mg of MAC can be reused up to five times without any loss of recovery for cadmium adsorption.

Effect of coexisting ions

The existence of coexistence ions may affect the preconcentration of cadmium by competitive adsorption. Therefore, the effect of common elements on the recovery of Cd(II) ions was investigated. An ion was considered to interfere when its presence produced a variation in the absorbance of the sample by more than 5%. The results given in Table 3 show that various potentially interfering ions in 100 fold excess had no obvious influence on the recovery of 100 ng L^{-1} Cd(II) ions under the optimum conditions. Therefore, the recommended method is suitable for preconcentration and determination of cadmium in various water samples.

Analytical figures of merit

Under the optimum conditions, the calibration curve was linear in the range of 10-700 ng L^{-1} of cadmium. The detection limit based on three times of the blank signals (n=8) was 4 ng L^{-1}. The relative standard deviation (RSD) for five replicate analysis of 100 ng L^{-1}cadmium was found to be 3.5%. The enrichment factor, calculated as the ratio of the slope of calibration curve for cadmium submitted to the recommended extraction procedure and that obtained without the preconcentration step, was 150. Comparison of the proposed method with other approaches in the literature for preconcentration and determination of cadmium is given in Table 4. Generally, this method is comparable to those of reported methods and exhibits relatively high enrichment factor and very low detection limit.

Analytical applications

The accuracy of the developed method was evaluated by the analysis of a certified reference material, JR-1. The determined value of 25±0.9 ng g^{-1} was in good agreement with the certified value of 26 ng g^{-1}. The method was applied to determine cadmium in different water samples. The analytical results, along with the recoveries for the spiked samples, are presented in Table 5. As shown, the proposed method is reliable for determination of cadmium in environmental water samples.

4. Conclusion

In this work, synthesized magnetic activated carbon nanocomposite as a solid phase extractor was employed for separation/preconcentration of Cd(II) ions at ultratrace levels in water samples. The MAC was prepared from carrot dress with low cost and shows very high adsorption capacity without any harm to environment. These nanoparticles are magnetically separable due to their superparamagnetic characteristics and therefore makes the SPE method time saving. The proposed preconcentration procedure was validated using a certified reference material. The method provides high enrichment factor and low detection limit and is suitable for cadmium determination with good accuracy in aqueous samples when dealing with large volumes of sample solutions and complex matrixes.

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