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Preconcentration of Copper Ion on Modified Magnetic Fe_3O_4@SiO_2 Nanoparticles in some Environmental Samples and its Determination by Electrothermal Atomic Absorption Spectrometry

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A B S T R A C T

Magnetic silica nanoparticles modified by 3-[2-(2-aminoethylamino) ethylamino] propyl-trimethoxysilane (AAAPTS) and Salicylaldehyde were prepared and used as a new adsorbent for extraction and preconentration of copper ion through magnetic solid-phase extraction (MSPE) method. After adsorption, these ions were desorbed with nitric acid followed by the determination with electrothermal atomic absorption spectrometry. Fourier transform infrared spectroscopy (FT-IR), field emission scanning electron microscopy (FE-SEM), transmission electron microscopy (TEM), X-ray diffraction (XRD) spectrometry and vibrating sample magnetometer (VSM) were used to characterize the adsorbent. The MSPE conditions were optimized.

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INTRODUCTION

Among nano-materials magnetic nanoparticles are main interests of many researchers due to their worthy magnetic properties. Magnetic nanoparticles have a wide range of applications, including catalysts [1], biomaterial/biomedicine [2], photocatalysts [3], electrochemical and bioeletrochemical sensors [4], microwave absorption [5], magnetic resonance imaging [6], medical diagnosis, material used for data storage [7], bioremediation [8] and modified magnetic copper ions for environmental case studies. While talking of, various magnetic nanoparticles, modified magnetite (Fe_3O_4) have been used for a number of applications due to its super paramagnetic properties. These modified nanoparticles not only protect the magnetic nano particles, but, also enhanced and provided a new platform for further functionalization that enhances the properties of the novel developed modified magnetic nanoparticles. With wide application of magnetic nanoparticles in energy saving process and nanofluids one can find the product is useful for energy and environmental friendly processes.

Copper is one of the most widely distributed elements in the environments of industrialized countries [9]. Generally, metals are potentially toxic to living organisms at variable concentrations. Heavy metals and metalloids that are not necessary for metabolic activities, consider being toxic to the cell even at quite low concentrations. The environmental exposure to the heavy metal ions is a well-known risk factor for human, plants and animals health [10-12]. Electrothermal atomic absorption spectrometry (ETAAS) is a good technique for the determination of ultra-trace amounts of heavy metals in several types of samples due to its sensitivity. Its particular advantages, such as the reduced sample solution requirements and the possibility to carry out in situ sample decomposition inside the graphite furnace are the mainattributes, which turn ETAAS into the technique of choice for such samples [13, 14]. However, due to its high sensitivity, ETAAS is sparingly used for copper determinations in samples [15, 16].

Recently, a novel SPE procedure, based on the use of magnetic or magnetizable materials called MSPE, has also been developed. A distinctive superiority of this

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technology is that solid-liquid separation can be easily achieved by the action of an external magnetic field instead of tedious and time-consuming centrifugation or filtration procedures which simplify the sampling and collection processes. Obviously, the best adsorbents must have high specific surface area, good selectivity, suitable chemical and physical stability was used in this work. However, the direct determination of trace concentration of copper in the presence of relatively high concentration of other diverse ions is a difficult task. Hence, separation and preconcentration steps are necessary prior to the copper detection, particularly when they exist at trace levels of concentration. Solid-phase extraction (SPE) is the most common technique because it offers many practical and operating advantages such as simple operation, high extraction efficiency, rapid phase separation and the ability of combination with different detection techniques. In SPE procedure, the choice of appropriate adsorbent is a critical factor to obtain full recovery and high selectivity. Then, this adsorbent was applied in magnetic solid-phase extraction (MSPE) of Cu (II) prior to electrothermal atomic absorption spectrometric (ETAAS) determination.

MATERIAL AND METHODS

A Stock solution of copper was prepared by dissolving of Cu $(NO_3)_2.3H_2O$ (Merck, Darmstadt, Germany) into deionized water. Working solutions were prepared daily by appropriate dilution of stock solutions. $FeCl_3.6H_2O$, $FeCl_2.4H_2O$, NH_3, ethanol andHNO_3were analytical grades and purchased from Merck Company. Tetraethyl orthosilicate (TEOS) for preparing silica shell on magnetic nanoparticles was also obtained from Merck. 3-[2-(2-aminoethylamino) ethylamino]propyl-trimethoxysilane (Acros organics,New Jersey, USA) and Salicylaldehyde were used to prepare the modified adsorbent in this study obtained from Merck.High purity reagents from Sigma (St. Louis, MO, USA) were used for studying interference effects.

The concentration of Cu (II) was determined by electrothermal atomic absorption spectrometry using a model Spectra AA 220 apparatus (Varian, Victoria, Australia).Optimum operating parameters for them are given in Table1. The samples were weighed using an electronic balance Mettler AE-160 (Greifensee, Switzerland). The pH measurements were carried out with a Metrohm 827 pH-meter (Herisau, Switzerland) supplied with a combined glass–calomel electrode. Magnetic stirrer hot plate and mechanical stirrer (2000 rpm) and oven model 100 (Memmert, Frankfurt, Germany) were used to homogenize. To disperse the nanoparticles in solutions, a Sonorex digitec model DT 225H with 35 KHz ultra-sonication(Bandelin, Berlin, Germany) was used. Fourier transform infrared (FT-IR) spectra were taken using a Tensor 27 spectrometer (Bruker, Saarbrucken, Germany). Field emission-scanning electron microscopy (FE-SEM) images were obtained on a model Hitachi S-4160 (Tokyo, Japan). AnLEO 912AB transmission electron microscope (TEM),(Carl Zeiss Inc., Jena, Germany) was used. The powder X-ray diffraction (XRD) patterns were examined on a model X'PertPro diffractometer (Panalytical, Almelo, The Netherlands) using Cu Kα radiation in the 2ɵ range 10–80°. Magnetic measurements were carried out using a vibrating sample magnetometer (VSM) model MDKFD (Danesh Pajohan Kavir Co. Kashan, Iran).

Preparation of Fe_3O_4 magnetic nanoparticles

The preparation of Fe_3O_4 magnetic nanoparticles were based on dissolving $FeCl_3.6H_2O$ (11.68 g) and $FeCl_2.4H_2O$ (4.30 g) in 200 mL deionized water at 80 ºC with a mechanical stirrerspeed of 1000 rpm for 30 min. Nitrogen gas was continually bubbled through this solution to expel oxygen. Following that, 25 mL of 25% NH_3 was rapidly added to the solution. After that, the color of the bulk solution changed from orange to black immediately. The magnetite precipitates were collected by a magnet after washing several timeswith deionized water and then with ethanol. Finally, they were dried in the oven for 18 hours at 80 ºC.

SiO_2 coating of the Fe_3O_4 magnetic nanoparticles

To protect the magnetic core by the silica film and prepare core–sell magnetic nanoparticles, the as-prepared Fe_3O_4 (0.5 g) were dispersed in a solution composed of 80 mL ethanol and20 mL double distilled water by sonicating for 30 min. Then, 5 mL ammonia solution (25 wt%) and 4 mL TEOS were added sequentially. The resulting suspension was stirred and allowed to react for 24 h at room temperature. Finally, the product was collected by a magnet, rinsed with deionized water, ethanol and dried in the oven for 8 hours at 80 ºC.

Surface modification of core–shell Fe_3O_4@silica with AAAPTS and salicylaldehyde

The core–shell Fe_3O_4@silica nanoparticles (600 mg) were suspended in 80 mL of dry toluene under a nitrogen atmosphere. After the addition of AAAPTS (4 mL), the mixture was refluxed for 24 h at 80 °C. The resulting product,asan adsorbent, was collected with a magnet, washed with dry toluene, deionized water andethanol and then dried at room temperature. 0.5 g amount of the Fe_3O_4@SiO_2@AAAPTS with 2 mL Salicylaldehyde and 40 mL methanol and poured into the balloon for 24 h were synthesized and separated by a magnet. And the ions were washed several times with water and then placing was dried at room temperature for 24 hours.

Magnetic solid-phase extraction procedure

MSPE was carried out as follows. A portion of 25 mL sample solution containing Cu (II) was transferred into a

100 mL glass flask and the pH was adjusted to 4.0 using HCl solution. Then, 250 mg of the synthesized Fe_3O_4@SiO_2@AAAPTS modified was added and the mixture was ultrasonication for 5 min. In this step, the analyte was adsorbed on the adsorbent. Then, a piece of a magnetwas placed outside the flaskto collect adsorbent and the supernatant was decanted directly. In desorption step, 3.0 mL HNO_3 (4.0 mol L^{-1}) was added as eluent and ultrasonication for 5 min. Finally,the magnet was used again to collect the nanoparticles, and the eluent was transferred into a test tube for subsequent ETAAS analysis.

RESULTS AND DISCUSSION

FT-IR spectroscopy

The FT-IR spectrum of naked Fe_3O_4MNPs shows the absorption peak at 562 cm^{-1} which correspond to the Fe–O vibration of bulk magnetite Fig.1A.The silica shell coated on the surface of Fe_3O_4 core can be confirmed by the spectrum shown in Fig. 1B. The bands at 1092 cm^{-1} and 799 cm^{-1} are assigned to Si–O–Si vibrations, the peak at 948 cm^{-1} was related to the vibration of Si–OH group and the absorption peak of Fe_3O_4 at 573 cm^{-1} indicated that MNPs is covered by silica layer through experiment.In the spectrum of AAAPTS Fig. 1C, the peaks at 3292 cm^{-1} and 1593 cm^{-1} can be assigned to N–H stretching and bending vibrations, respectively. The two peaks at 2937 cm^{-1} and 2839 cm^{-1} are due to asymmetrical and symmetrical C–H stretching vibrations and C–H bending peak is observed at 1462 cm^{-1}. The band at 1194 cm^{-1} is ascribed to the stretching of the C–N stretching bonds.Another two peaks at 1082 cm^{-1} and 819 cm^{-1} might be ascribed to Si–O–Si vibrations. Imine peak is observed at 1633 cm^{-1}.Also, the successful surface modification of Fe_3O_4@SiO_2 nanoparticles can be confirmed by FT-IR that shows in Fig. 1E.

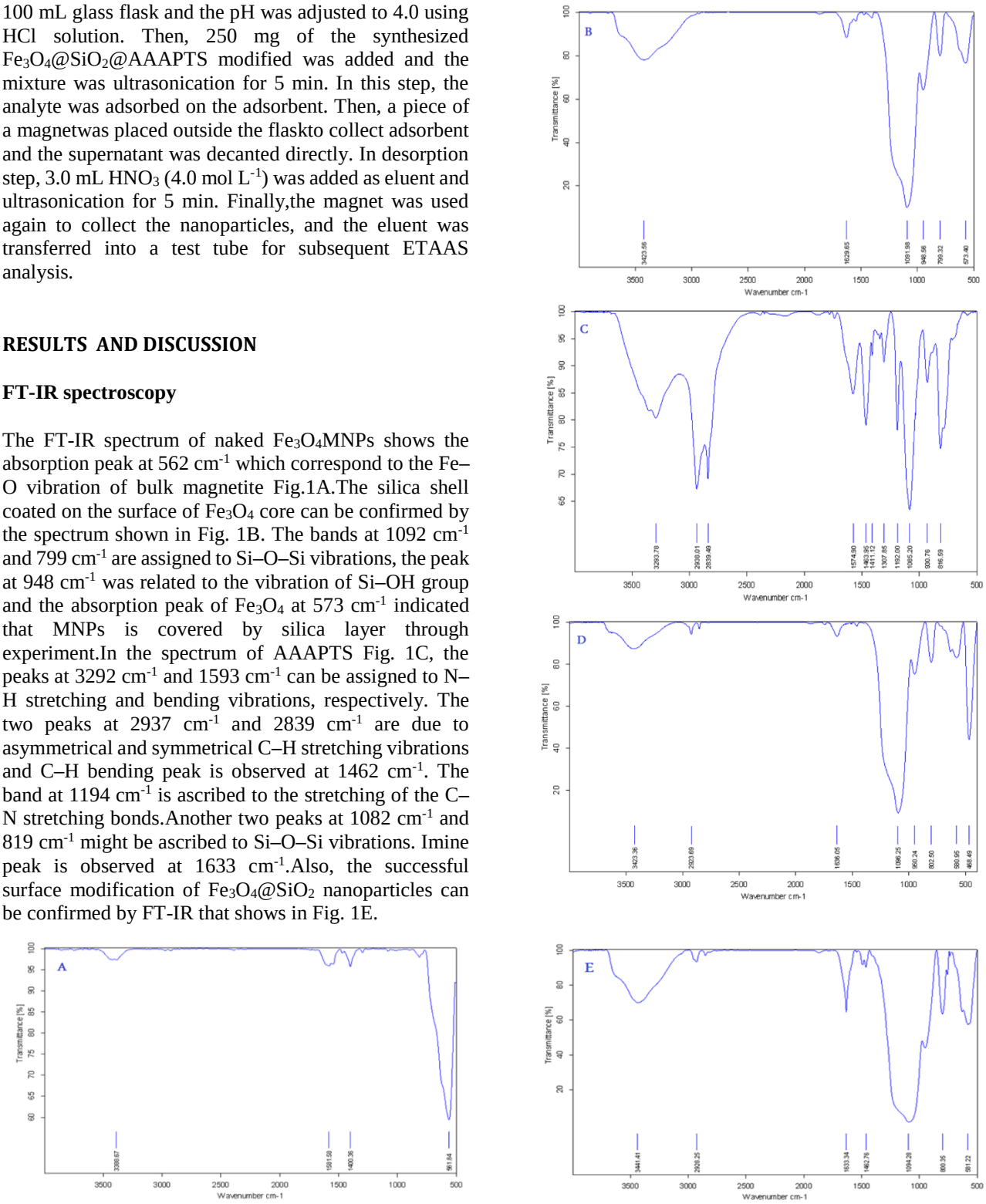
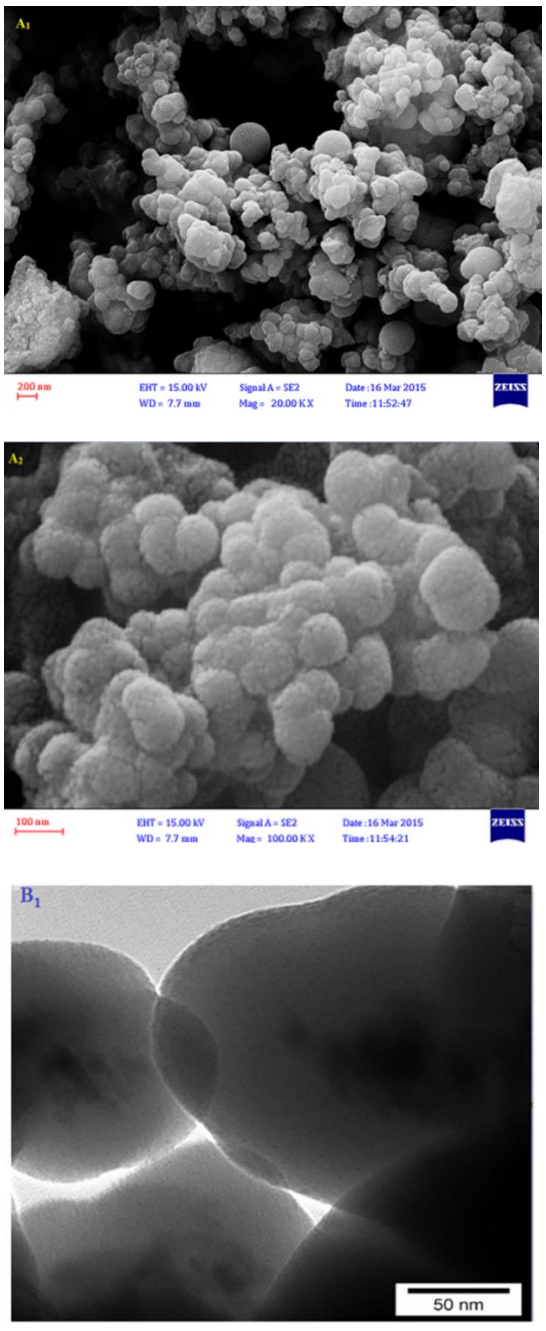


Figure 1. FT-IR spectra of (A) Fe_3O_4, (B) Fe_3O_4@SiO_2, (C) AAAPTS (D) Fe_3O_4@SiO_2@AAAPTS and (E) Fe_3O_4@SiO_2@AAAPTS modified nanoparticles.

TEM and FE-SEM images

The morphological feature of the synthesized Fe_3O_4@SiO_2@AAAPTS nanoparticles were characterized by FE-SEM and TEM images and shown in different magnification Fig. 2.TEM images show that MNPs nanoparticles had entrapped in the silica sell successfully, in which the diameter of the magnetic core is lower than50 nm.



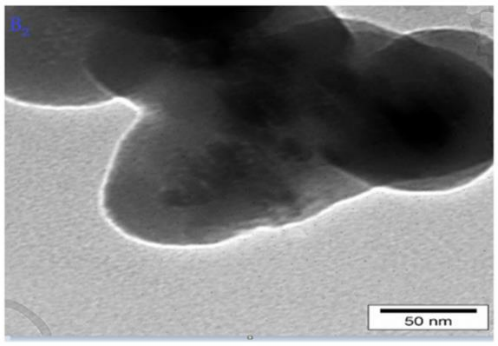


Figure 2. FE-SEM (A_1&A_2) and TEM (B_1&B_2) images of the nanoadsorbent particles.

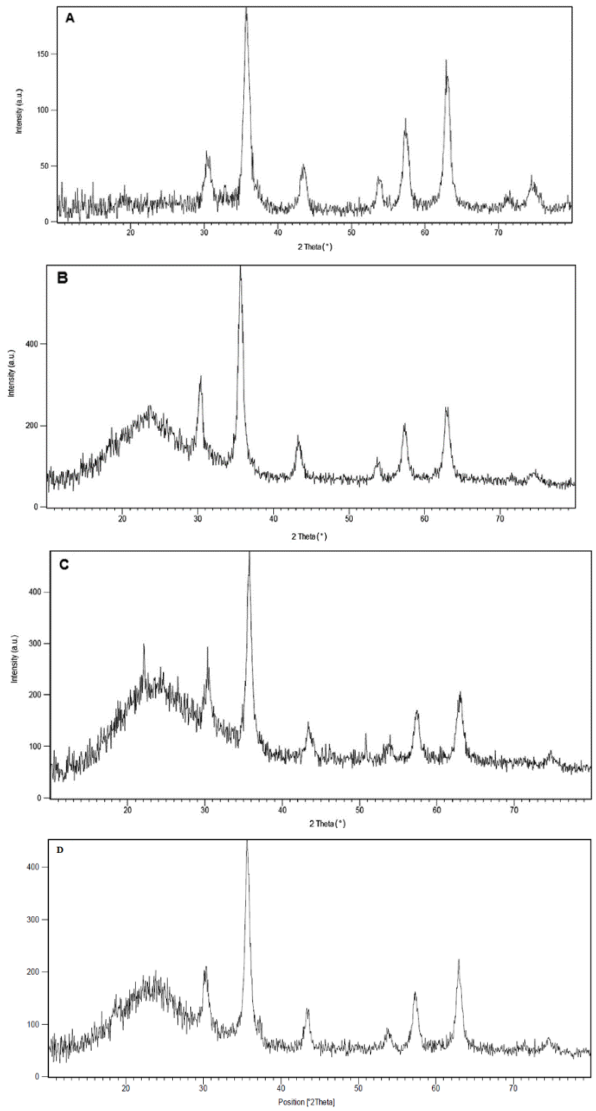


Figure 3. XRD patterns of (A) Fe_3O_4, (B) Fe_3O_4@SiO_2 and (C) Fe_3O_4@SiO_2@AAAPTS (D) Fe_3O_4@SiO_2@AAAPTS modified nanoparticles.

XRD patterns

A typical XRD pattern of the synthesized Fe_3O_4, Fe_3O_4@SiO_2 and Fe_3O_4@SiO_2@AAAPTS modified nanoparticles are shown in Fig. 3. The diffraction peaks such as (2 2 0), (3 1 1), (4 0 0), (4 2 2), (5 1 1) and (4 4 0), can be indexed to face-centered cubic structure of magnetite. Also, board peak at low diffraction angle which corresponded to the amorphous peak of SiO_2 was displayed in Fig. 3B. Salicylaldehyde and AAAPTS modified Fe_3O_4@SiO_2 surface can be confirmed from the XRD data.

Magnetic measurements

Fig. 4 shows that the saturation magnetization values were 72.3, 44.1, and 34.6 emu g^{-1} for Fe_3O_4, Fe_3O_4@SiO_2 and Fe_3O_4@SiO_2@AAAPTS modified, respectively. Compared to the Fe_3O_4, the saturation magnetization of Fe_3O_4@SiO_2@AAAPTS modifiedreduced to 34.6 emu g^{-1}, but it is sufficient for magnetic separation with a conventional magnet.

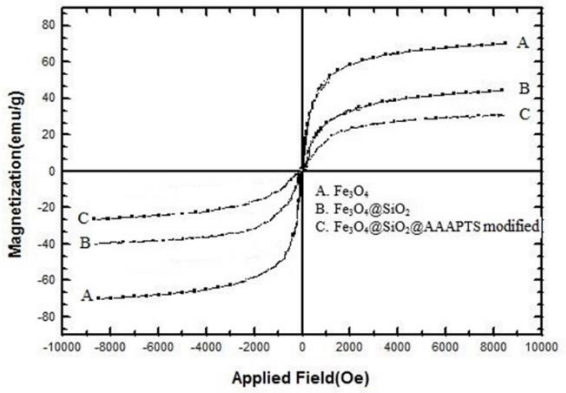


Figure 4. VSM curves of (A) Fe_3O_4, (B) Fe_3O_4@$SiO_2$and (C) Fe_3O_4@SiO_2@AAAPTS modified nanoparticles.

Optimization of extraction conditions

Several experimental variables affecting the extraction recovery of bismuth and lead ions were investigated, including, sample pH, the eluting solvent,the adsorbent amount,ultrasonic time and sample volume. In all optimizationexperiments,the one-at-a-time strategy was used.

Effect of pH

The sorption ability of copper ion by the as-prepared modified MNPs is significantly dependent on the pH value of the aqueous solution. The influence of pH on the extraction recovery of target ions was tested in the range 2 to 7 when the other parameters were kept constant. Fig. 5 shows the recovery percentage increased along with increasing pH value from 2–4 and maximum recovery was obtained at pH 4. When the pH was further enhanced from 4 to 7, the recovery percentage decreased silently. Therefore, pH 4 was selected in the following experiments.

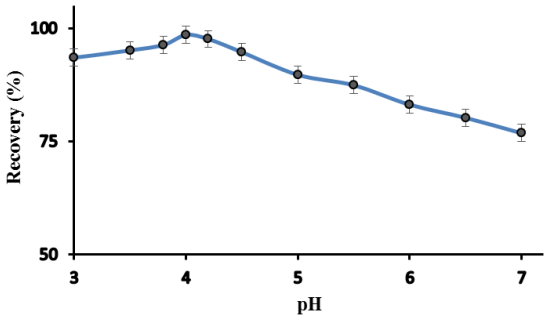


Figure 5.Effect of pH on the extraction efficiency of copper.
Experimental conditions were the following: 0.025 g sorbent, Eluent; 3.0 mL 4.0 mole L^{-1} HNO_3, ultrasonication time; 5 min, Cu(II); 0.15 ng mL^{-1}, (N =3).

Effect of eluent

The effect of type and volume of eluent on the recovery of Cu (II) was investigated. The sorption of these ions on the adsorbent at an acidic solution is low. It means that desorption can be done in this media. Therefore, various acidic solutions were used as eluent and the results showed that HNO_3 with the concentration of 4 mol L^{-1} was the best ones. Also, the eluent should elute analytes quantitatively in a small volume. Thus, the volume range of 0.5–5 mL was tested and finally, 3.0 mL of 4 mol L^{-1} HNO_3 was chosen for the elutionFig. 6 and 7.

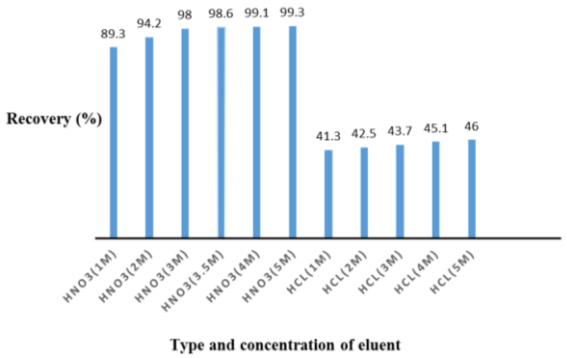


Figure 6. Effect of type and concentration of eluent on the extraction efficiency of copper, Experimental conditions were same as Fig. 5. except of pH was 4.0.

Effect of Fe_3O_4@SiO_2@AAAPTS modified amount

In the MSPE technique, the amount of magnetic adsorbent is one of the main factors affecting on the extraction of the analytes. Hence, this parameter was studied in the range 0.005–0.1 g. the results indicate the recovery percentage increased with an increase in the adsorbent content which could be due to the availability

of more sorption sites and larger surface area. Up to a certain value (0.025 g), no further increase in percent occurred as an increase in adsorbent content. So, 0.025 g was used in the all subsequent experiments Fig. 8.

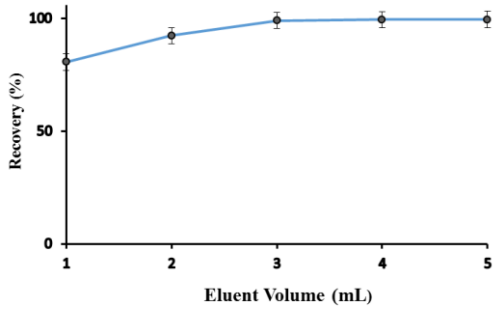


Figure 7. Effects of eluent volume on the extraction efficiency of copper, Experimental conditions were same as Fig. 5. except of pH was 4.0.

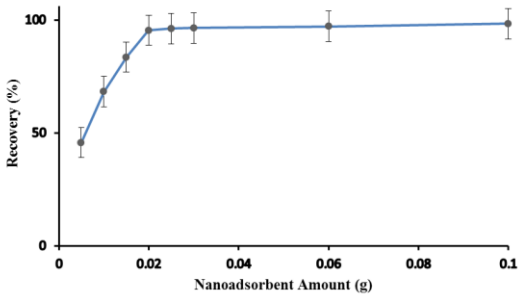


Figure 8. Effects of nanoadsorbent amount on the extraction efficiency of copper, Experimental conditions were same as Fig. 5. except of pH was 4.0.

Effect of ultrasonication time

In this method, to obtain adsorption and desorption times, ultrasonic was used. For both adsorption and desorption, ultrasonic times in the range 1–20 min were optimized. The experimental results indicated that 5 min was sufficient for achieving quantitative recovery of bismuth and lead ions for both adsorption and desorptionFig. 9.

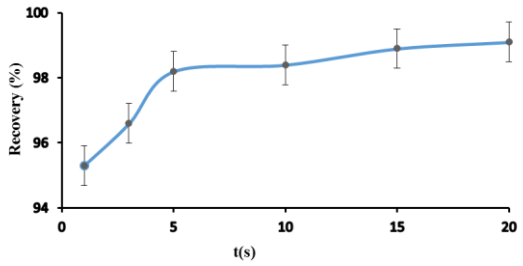


Figure 9. Effects of ultrasonication time on the extraction efficiency of copper, Experimental conditions were same as Fig. 5. except of pH was 4.0.

Effect of sample volume

The sample volume is an important parameter to obtain a high enrichment factor. Experimental analysis showed that when sample volume was 700 mL, quantitative recovery was obtained and theoretical enrichment factor was 233 for target ion Fig. 10.

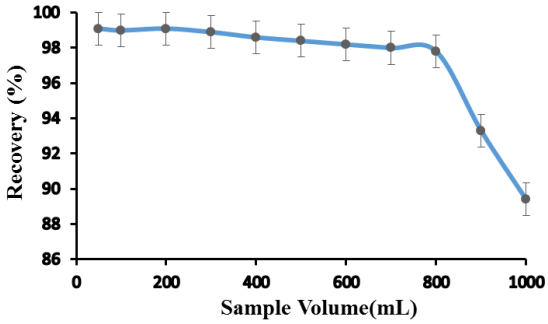


Figure 10. Effects of sample volume on the extraction efficiency of copper, Experimental conditions were same as Fig. 5. except of pH was 4.0.

Adsorption capacity

The adsorption capacity is an important factor to evaluate the Fe_3O_4@SiO_2@AAAPTS modified nanoadsorbent. It is the maximum analytes quantity taken up by 1.0 g of synthesized nanoadsorbent. In order to determine this factor, 50 mg of the nanoadsorbent were subjected to several loadings with 25 mL sample solution containing Cu(II) with pH = 4.0 and then, followed by the determination of retained analytes using ETAAS. The values of adsorption capacity increased with the increase of initial concentrations of target ion and then it reached a plateau. The static adsorption capacity 14.7 mg g^{-1} (14.7 mg copper in per gram of nanoadsorbent) of the nanoadsorbent was obtained.

Effect of coexisting ions interference

Under optimized conditions, the interference of various cations and anions on the recovery of copper ion was investigated. Results obtained showed that Fe_3O_4@SiO_2@AAAPTS modifiedis a good candidate to preconcentration of target analytes without any serious matrix interference in different real samples (Table 2).

Analytical performance

Analytical performance such as linear range (LR), limits of detection (LOD) and repeatability were studied under the optimized working conditions. The obtained linearity was 0.004–0.250 ng mL^{-1} for Cu (II). The LOD (defined as $3Sd_{blank}/m$, where Sd is the standard deviation of the blankreadings for ten replicate analysis and m is the slope of the calibration curve) was 1.2 ng L^{-1} copper. Also, the resultant repeatability expressed as relative standard deviations (RSD, N=7) was calculated as 3.0% for 0.15 ng mL^{-1} concentration of Cu (II).

Analysis of the real samples

The method was applied to the determination of trace amount of Cu(II) in a variety of samples. The samples were also analyzed after spiking with different concentrations of copperion with high recoveries. Tap water, well water, and human hair samples were analyzed. The analytical results are presented in Table 4. The results show that the method is suitable for the analysis of real samples.

Comparison with other methods

A comparison of the method with other preconcentration procedures is given in Table 3. As seen from the data in this table, the method using Fe_3O_4@SiO_2@AAAPTS modified has high enrichment factor and relatively low LOD, wider linear range and lowers RSD that it is comparable or better than ones reported elsewhere [17-22].

CONCLUSION

Fe_3O_4@SiO_2@AAAPTS modified nanoparticles with good saturation magnetization have been successfully prepared and used as a novel and effective adsorbent for extraction and preconcentration of a copper ion from aqueous solutions prior to a determination by ETAAS. It is a very fast extraction method, because of a high surface area of the adsorbent and fast magnetic separation. Ease of operation, high selectivity and sensitivity are other advantages. Also, the combination of MSPE by the Nano adsorbent with ETAAS offers significant analytical performance. The obtained results show that this method is suitable to determine copper ion by ETAAS in several types of natural water, hair samples at ultra-trace levels.

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Persian Abstract

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چکیده

نانوکامپوزیت مغناطیسی اصلاح شده با ۳-[۲-(۲-آمینواتیل‌آمینو) اتیل‌آمینو] پروپیل- تری‌متوکسی‌سیلان و سالیسیل آلدهید تهیه و به عنوان جاذب جدید برای استخراج و پیش تغلیظ یون مس، با استفاده از روش استخراج فاز جامد مغناطیسی مورد استفاده گرفت. بعد از جذب، یون های واجذب شده با نیتریک اسید توسط اسپکتروسکوپی جذب اتمی کوره گرافیتی اندازه گیری شد. ازدستگاه های طیف‌سنجی مادون قرمز، میکروسکوپالکترونیروبشی، میکروسکوپالکترونیعبوری، پراش پرتو ایکس، مغناطیس‌سنج نمونه ارتعاشی جهت مشخصه یابی جاذب مورد استفاده قرار گرفت. که شرایط استخراج فاز جامد مغناطیسی بهینه شد.