

Determination of Copper and Zinc Ions by Flame-AAS After Preconcentration Using Sodium Dodecyl Sulfate Coated Alumina Modified with 3-((1*H*-Indol-3-yl)-3,4,5-trimethyl)-1*H*-indole

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A sensitive and simple method for the simultaneous preconcentration of trace amount Cu^{2+} and Zn^{2+} ions in some real samples has been established, which is based on the sorption of Cu^{2+} and Zn^{2+} on 3-((1*H*-indol-3-yl)-3,4,5-trimethyl)-1*H*-indole (ITMI) loaded on sodium dodecyl sulfate (SDS) coated alumina. The metal absorbed on the complexes was eluted using 3 mol/L nitric acid. The influences of the analytical parameters including pH and sample volume were investigated. The effects of matrix ions on the retentions of the analytes were also examined. The recoveries of analytes were generally higher than 95% with a low RSD. The method has been successfully applied to content evaluation of these metals in real samples.

Keywords surfactant coated alumina, atomic absorption spectrometry, solid phase extraction

Introduction

Due to negative effects of heavy metal ions on human health, heavy metal pollution in environment is one of the important problems for human.¹⁻³ The determination of traces of heavy metal ions in environmental samples is restricted by two main difficulties: the very low concentration of heavy metal ions, which may be lower than the detection limit of many analytical techniques including flame atomic absorption spectrometry (FAAS) and the interfering effects of the matrix. In order to achieve accurate, reliable, and sensitive results, preconcentration and separation are needed when the concentrations of analyte metal ions in the original material or the prepared solution are too low to be determined directly by FAAS.⁴⁻⁷ In this regard, various supports in our laboratory were recommended for the enrichment and separation of heavy metal ions⁸⁻¹² as the most useful ways to concentrate trace heavy metals, or to separate analytes from a sample matrix.

Solid-phase extraction (SPE) technique has increasingly become popular in comparison with the more traditional liquid-liquid extraction methods because of its several major advantages such as: (i) simplicity to operate; (ii) high preconcentration factor; (iii) rapid phase separation; and (iv) the ability of combination with different detection techniques.

Among solid-phase adsorbents, SDS coated alumina is very useful for preconcentration and separation of

metal complexes¹³⁻²⁸ because of its good physical properties such as its porosity, uniform pore size distribution, high surface area as chemical homogeneous non-ionic structure, and good adsorbent properties for great amounts of uncharged compounds.

Generally, the design of a stable and selective solid phase sorbent for separation and preconcentration of a target metal ion depends on different factors related to the nature of solid support, its surface area and activity as well as other important factors related to the organic complexing agent bound to the solid substrate. Recent studies on the use of surfactant-coated mineral oxide columns for SPE have demonstrated that these new sorbent materials are a promising tool for the extraction/preconcentration of organic compounds in a wide polarity range.

The purpose of this work is to investigate the feasibility of adsorption of these ions including, Cu^{2+} and Zn^{2+} ions on SDS coated alumina modified with ITMI. For obtaining maximum signal for evaluation of these ion contents, the effective parameters *viz.* pH of sample, amount of ITMI and solid phase, type of eluting agent and flow rate were optimized.

Experimental

Instrument

The measurements of metal ions were performed

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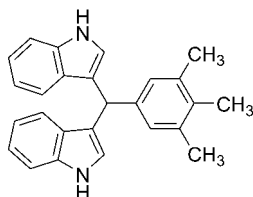
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with a Shimadzu 680 AA atomic absorption spectrometer equipped with a hollow cathode lamp and a deuterium background corrector, at respective wavelengths (resonance line) using an air-acetylene flame. The instrumental parameters were those recommended by the manufacturer. A Metrohm 691 pH/Ion meter with a combined glass-calomel electrode was used for adjustment of test solution pH.

Reagent and solution

Acids and bases were of the highest purity available from Merck and used as received. Doubly distilled deionized water was used throughout. Nitrate salts of lead, cadmium, mercury, cobalt, nickel, copper, zinc, magnesium, calcium, strontium, barium, silver, sodium and potassium (all from Merck) were of the highest purity available and used without any further purification. The pH adjustment was done by addition of dilute nitric acid or potassium hydroxide to prepare the desired pH solution. The γ -alumina with mesh in range of 20–60 was purchased from Merck Company. The ligand 3-((1*H*-indol-3-yl)-3,4,5-trimethyl)-1*H*-indole (Scheme 1) was synthesized according to the literature.²⁹

Scheme 1 Structure of ligand



Preparation of ITMI coated alumina

20 mg of SDS and 25 mg of ITMI were added to 40 mL of water solution containing 1 g of alumina particles and the pH was adjusted to 10 by addition of dilute potassium hydroxide. The pH was adjusted to 2 with 2 mol·L⁻¹ hydrochloric acid to form ITMI-impregnated ad-micelles on alumina particles while shaking the suspension with a stirrer. After mixing for 15 min, the supernatant solution was discarded and the remaining was packed into a column. The column was washed by passing 6 mL of 3 mol·L⁻¹ HNO₃, and then the column was neutralized with KOH. When kept in a refrigerator the sorbent was stable at least for one week. The concentration of SDS was fixed below the critical micellization concentration (CMC) (8 × 10⁻³ mol/L) of SDS.

Test procedure

The pH of model solutions containing analytes was adjusted to pH 2–9 by dilute nitric acid or potassium hydroxide. The samples were passed through the column at a flow rate of 3 mL·min⁻¹, to effect the deposition of analyte. The adsorbed ions were then eluted with 8 mL of 3 mol·L⁻¹ nitric acid. The analyte concentrations in the eluent were determined by flame atomic absorption spectrometry.

Pretreatment of real samples

Leaves of lettuce tree, radish, eucalyptus and nettle were purchased from Firouzabad, Iran. Afterwards, they were dried and taken in small meshes. 40 g leaves of spinach was heated in a silica crucible for 3 h on a hot plate and the charred material was transferred to a furnace and heated overnight at 650 °C. The residue was cooled, treated with 10.0 mL of concentrated nitric acid and 3 mL of 30% H₂O₂, and again kept in the furnace for 2 h at the same temperature so that no organic compound traces were left. The final residue was treated with 3 mL of concentrated hydrochloric acid and 2–4 mL of 70% perchloric acid and evaporated to fumes, so that all the metals were changed to respective ions. The solid residue was dissolved in water, filtered and keeping the pH at 6.0 made up to 250 mL by addition of diluted KOH. Then the preconcentration procedure given above was applied.

A 20 g sample of cucumber, rice and tomato was heated in a silica crucible for 3 h on a hot plate and the charred material was transferred to a furnace for overnight heating at 650 °C. The residue was cooled, treated with 10.0 mL of concentrated nitric acid and 3 mL of 30% H₂O₂ and again kept in the furnace for 2 h. The final residue was treated with 3 mL concentrated hydrochloric acid and 2 to 4 mL of 70 wt% perchloric acid and evaporated to fumes. The solid residue was dissolved in water, filtered and the pH was adjusted to 6.0 by the addition of KOH and HCl. The preconcentration procedure given above in Section “Preparation of ITMI coated alumina” was then applied to these solutions.

Menthe sativa, borage (cowslip), rose water and detergent samples were collected from Omidyeh, Iran. Before the analysis, the samples were filtered through a cellulose membrane filter (Millipore) of pore size 0.45 μm. The organic contents of the water samples were oxidized in the presence of 1% H₂O₂ by addition of concentrated nitric acid. After acidification to 1% with concentrated nitric acid, they were stored in polyethylene bottles. For the preconcentration procedure, pH of the 250 mL of water samples was adjusted to 6 and the sample passed through the column at a flow rate of 3 mL·min⁻¹. The metals retained on modified solid phase were eluted with 8 mL of 3 mol·L⁻¹ HNO₃ in acetone.

Results and discussion

In order to avoid micelle formation to prevent from adsorption of target sample and ligand on support, SDS concentration was held lower than its CMC. The adsorption of SDS is highly dependent on the solution pH. Negatively charged SDS was more effectively adsorbed on the positively charged alumina surfaces at lower pH; nearly complete adsorption was achieved in pH 1–4 by shaking for 15 min. About 99% of SDS was adsorbed even in 1 mol·L⁻¹ nitric acid. Activated alumina can function as either a cation or as an anion preconcentrator depending on the pH. Acid pH values have been rec-

ommended for adsolubilization of organic compounds on SDS-coated γ -alumina. The adsolubilization capability of this sorbent greatly increases by decreasing the pH as a result of the higher charge density on the mineral oxide surface, *i.e.* the maximal amounts of adsorbed SDS in the hemimicellar and ad-micellar regions at pH 2 was 7-fold greater than that at pH 6, respectively. In fact, modification of alumina surface via physical loading of organic substrates is still attractive due to its great simplicity. New sorbents may be obtained with quite strong hydrogen bonding if we take into consideration the strength of proton-acceptor nature of the selected organic modifier. Ligand is expected to fulfil this requirement. It has a polar nature and quite strong hydrogen bond acceptor, in addition to its small and compact structure. When it is free (not bind to a solid support), it can coordinate with hard and soft metal ions via its own O and S donor sites.

Influence of pH

One of the most important factors in a solid phase extraction procedure is the pH of the aqueous phase for the quantitative recoveries. The reaction between analyte ions and the complexing agent can be influenced by changing the pH value. The influences of the pH on Cu^{2+} and Zn^{2+} ion complexation with ITMI and their sorption onto SDS coated alumina were investigated over the pH range from 2.0 to 9.0 keeping other parameters constant. Figure 1 shows that Cu^{2+} and Zn^{2+} ions were effectively adsorbed at pH of 6.0.

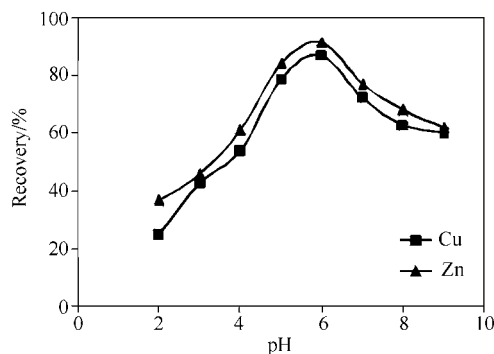


Figure 1 Effect of pH on analyte ion recovery ($N=3$).

The obtained results indicate that the ions could be retained quantitatively by the modified solid phase at the pH 7.0. The decrease in signal at $\text{pH} > 6.0$ is probably due to the precipitation of metal ions as their related hydroxide and the competition of hydronium ion toward complexation with ITMI, which led to the decrease in the recovery. To achieve high efficiency and good selectivity, a pH of *ca.* 6.0 was selected for subsequent work. In order to control the pH during the analytical procedure, it was adjusted to 6.0 by addition of KOH and HNO_3 .

Effect of amounts of ITMI

In order to determine the concentration of ITMI re-

quired for quantitative recoveries, the proposed method was applied. Firstly, some tests were carried out without the ligand at pH 6. The analyte ions were not quantitatively recovered without the ligand. Then the studies were performed with various amounts of the ligand in the range of 0–40 mg. It was found that, the recovery of the metal ions increased with increasing concentrations of ITMI added and reached a constant value (95%) with at least 25 mg. On this basis, studies were carried out at a ITMI quantity of 25 mg. This quantity of ITMI is enough for the separation-preconcentration procedure because of the very low level of the investigated metal ion concentrations in real samples. The sorbent exhibited the greatest affinity towards Cu^{2+} and Zn^{2+} ion extraction in all the metal ions.

Effect of amounts of solid phase

The influences of the amounts of solid phase with various compositions and amounts filled to the column were also investigated (Table 1). The results show that up to 0.6 g of solid phase, the efficiency of extractions increased with the increment of the solid phase. The effect of the amount of alumina on the sorption of metal ions at pH 6.0 was examined in the range of 0.1–1.0 g. Quantitative recoveries ($>95\%$) of Cu^{2+} and Zn^{2+} ions were observed using 0.6 g of solid phase. The recoveries of analytes above 0.6 g of alumina were below 95% with 8 mL of the eluent. In the proposed procedure, 0.6 g of solid phase was recommended. As it can be seen, synergic effect of presence of ligand, SDS and alumina is suitable for efficient and reproducible recovery of metal ions.

Table 1 Effect of amount of solid phase on recovery of metal ions ($N=3$).

| Amount of solid phase/g | Recovery/% | |
|---|------------|----|
| | Cu | Zn |
| 0.2 | 45 | 52 |
| 0.4 | 73 | 72 |
| 0.5 | 90 | 89 |
| 0.6 | 96 | 95 |
| 0.7 | 87 | 90 |
| 1 | 71 | 76 |
| 20 mg SDS, 0.6 g Al_2O_3 , without ligand | 59 | 67 |
| 0.6 g Al_2O_3 , 25 mg ligand, without SDS | 32 | 43 |
| 4 mg SDS, 0.1 g Al_2O_3 , 25 mg ligand | 48 | 58 |
| 12 mg SDS, 0.3 g Al_2O_3 , 25 mg ligand | 78 | 84 |
| 20 mg SDS, 0.6 g Al_2O_3 , 25 mg ligand | 93 | 96 |

Eluent types and eluent volume

The choice of a suitable eluent is very important for obtaining the successful and complete preconcentration and recovery of each ions. A satisfactory eluent should effectively elute the sorbed analytes with a small volume, which is needed for a high enrichment factor and

should not affect the accurate determination of the analytes and destroy life time and reusability of solid phase. The experimental results in Figure 1 show that the adsorption of these metal ions was negligible at $\text{pH} < 3$. For this reason, different acids and various concentrations and volumes of HNO_3 were attempted to be used as the eluent. Due to this point, various eluent solutions given in Table 1 were used for desorption of ions complexed with ITMI chelates from SDS coated alumina. Quantitative recoveries for Cu^{2+} and Zn^{2+} ions were obtained only with $3 \text{ mol}\cdot\text{L}^{-1}$ HNO_3 . Recoveries were not quantitative when other eluents were used in the present study. For this reason, HNO_3 was the best eluent and chosen as eluent and various concentrations and volumes of HNO_3 were attempted to elute the analytes retained in the modified column. The results obtained are given in Table 2. The volume of eluent is important for the high concentration factor. This was examined by varying of $3 \text{ mol}\cdot\text{L}^{-1}$ nitric acid volume to 8 mL from 10 mL. The smallest volume of $3 \text{ mol}\cdot\text{L}^{-1}$ nitric acid for the quantitative elution was defined as 6 mL. The effect of eluent volume on the recoveries of the analytes was also studied by using $3 \text{ mol}\cdot\text{L}^{-1}$ HNO_3 ; it was found that quantitative recoveries could be obtained with 8.0–10.0 mL of $3 \text{ mol}\cdot\text{L}^{-1}$ HNO_3 . Therefore, the volume of 8.0 mL of $3 \text{ mol}\cdot\text{L}^{-1}$ HNO_3 was used in the following experiments to obtain high enrichment factor.

Table 2 Effect of type and concentration of eluting agent on recovery of analytes

| Condition of eluting solution | Recovery/% | |
|---|------------|----|
| | Cu | Zn |
| H_3PO_4 ($4 \text{ mol}\cdot\text{L}^{-1}$) | 13 | 28 |
| H_2SO_4 ($4 \text{ mol}\cdot\text{L}^{-1}$) | 23 | 24 |
| HCl ($4 \text{ mol}\cdot\text{L}^{-1}$) | 82 | 76 |
| HNO_3 ($1 \text{ mol}\cdot\text{L}^{-1}$) | 41 | 46 |
| HNO_3 ($2 \text{ mol}\cdot\text{L}^{-1}$) | 63 | 59 |
| HNO_3 ($3 \text{ mol}\cdot\text{L}^{-1}$) | 93 | 94 |
| HNO_3 ($4 \text{ mol}\cdot\text{L}^{-1}$) | 89 | 93 |
| HNO_3 ($5 \text{ mol}\cdot\text{L}^{-1}$) | 80 | 95 |
| HNO_3 ($6 \text{ mol}\cdot\text{L}^{-1}$) | 76 | 85 |
| HNO_3 (2 mL, $3 \text{ mol}\cdot\text{L}^{-1}$) | 44 | 52 |
| HNO_3 (4 mL, $3 \text{ mol}\cdot\text{L}^{-1}$) | 60 | 67 |
| HNO_3 (6 mL, $3 \text{ mol}\cdot\text{L}^{-1}$) | 76 | 81 |
| HNO_3 (7 mL, $3 \text{ mol}\cdot\text{L}^{-1}$) | 86 | 88 |
| HNO_3 (8 mL, $3 \text{ mol}\cdot\text{L}^{-1}$) | 95 | 95 |
| HNO_3 (9 mL, $3 \text{ mol}\cdot\text{L}^{-1}$) | 95 | 96 |
| HNO_3 (10 mL, $3 \text{ mol}\cdot\text{L}^{-1}$) | 97 | 98 |

Amount of SDS

The concentration of surfactant aggregates (hemi-micelles, or ad-micelles) on mineral oxides mainly depends on the surfactant/oxide weight ratio. At surfactant concentrations higher than about 50 mg/g

alumina, a decrease in the percentage of ions retained was observed as a result of the formation of micelles (Figure 3). In the absence of SDS, ions were not retained on SDS-alumina. The retention of metal ions on hemi-micelles, which have a hydrophobic surface, was clearly dependent on analyte complex polarity. Therefore, addition of SDS is necessary. The formation of minute amounts of ad-micelles was essential to achieve complete adsolubilization of these ions as respective complex on the surface of support. At surfactant concentrations higher than about 50 mg/g alumina, a decrease in the percentage of ions retained was observed as a result of the formation of micelles.

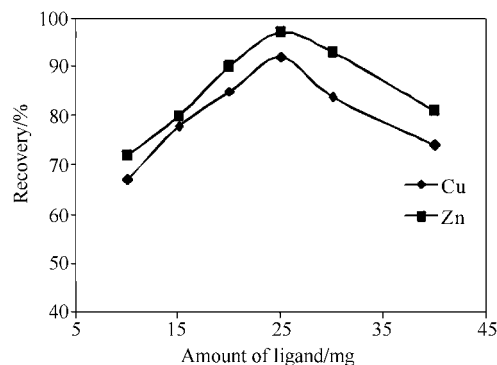


Figure 2 Effect of amount of ligand on analyte ion recovery ($N = 3$).

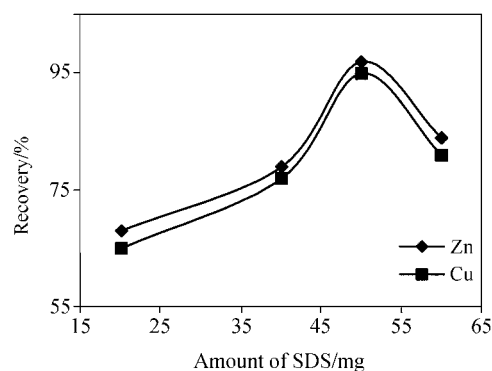


Figure 3 Effect of amount of SDS on ions.

Sample and eluent flow rate

The sample and eluent flow rates are important parameters to obtain quantitative retention and elution, respectively. The flow rates of the solutions were controlled by using the stopcock of the column. The influences of the sample and eluent flow rates on the recovery of metal ions were separately examined in the flow rates in the range of $1\text{--}5 \text{ mL}\cdot\text{min}^{-1}$. When the influences of the sample flow rates were examined, the eluent flow rate was kept constant as $1 \text{ mL}\cdot\text{min}^{-1}$. The retentions for the analytes were quantitative up to a sample flow rate of $3 \text{ mL}\cdot\text{min}^{-1}$. When the effects of the eluent flow rates were examined, the sample flow rate was fixed at $3 \text{ mL}\cdot\text{min}^{-1}$. In the eluent volume lower than 1.0 mL, because of insufficient eluent volume, the recoveries of the analyte ions were not quantitative. All

further studies were performed at $3 \text{ mL} \cdot \text{min}^{-1}$ flow rate for sample and $1 \text{ mL} \cdot \text{min}^{-1}$ for eluent solutions.

Effect of sample volume

Experiments to determine the sample loading volume were performed by passing increasing volumes between 250–2000 mL, at an increment of 250 mL between 250 and 1500 mL and at an increment of 50 mL between 1500 and 2000 mL of aqueous solutions at pH 7 containing $50 \mu\text{g}$ of all ions, through the 1 g modified solid phase column then determining the amount of all analyte ions eluted with nitric acid. This trait gives the feasibility of determination of the sample with different analyte concentration levels. However, it should be pointed out that the larger the amount of the sample solution, the longer the time (for passing it through solid phase) was required in the procedure. To trade off the enrichment factor and analytical speed, 1250 mL of sample and 8.0 mL of HNO_3 were used; therefore, an enrichment factor of 157 was obtained.

Effect of divers ions

To assess the possible analytical applications of the recommended procedure, the effect of some foreign ions, which interfere the determination of metal ions by the ITMI method or/and often accompany analyte ions in various real samples, was examined with the optimized conditions at above. For these studies, a fixed amount of metal ions was taken with different amounts of foreign ions and the recommended procedure was followed. The recoveries of metal ions in these studies were higher than 95%. Tolerable limit was defined as the highest amount of foreign ions that produced an error not exceeding 5% in the determination of investigated analyte ions by the combination of the column solid phase extraction and the flame atomic absorption spectrometric determination methods. The results are summarized in Table 3. As it is seen, large numbers of ions used have no considerable effect on the determination of analyte ions.

Table 3 Effects of the matrix ions on the recoveries of the examined metal ions ($N=3$)

| Interference | Recovery/% | | |
|------------------------------------|------------------------------------|----|----|
| | Interference to analyte fold ratio | Cu | Zn |
| K^+, Na^+ | 700 | 93 | 98 |
| $\text{HCO}_3^-, \text{SCN}^-$ | 700 | 93 | 98 |
| $\text{Al}^{3+}, \text{Ti}^{3+}$ | 1000 | 91 | 93 |
| Ni^{2+} | 500 | 90 | 94 |
| Co^{2+} | 500 | 91 | 92 |
| Cd^{2+} | 1000 | 92 | 95 |
| Fe^{3+} | 800 | 94 | 97 |
| $\text{Mn}^{2+}, \text{SO}_4^{2-}$ | 1000 | 90 | 92 |
| $\text{Hg}^{2+}, \text{Cl}^-$ | 300 | 92 | 96 |
| $\text{Ba}^{2+}, \text{Mg}^{2+}$ | 300 | 93 | 97 |
| Ag^+ | 800 | 91 | 95 |

Figures of merit

The sensitivity of the developed method is reflected by the limit of detection (LOD) studies, defined as the lowest concentration of metal ions below which quantitative sorption of the metal ion by basic alumina is not perceptibly seen. The detection limit (LOD) of the present work was calculated under optimal experimental conditions after application of the preconcentration procedure to blank solutions. The detection limits based on three times the standard deviations of the blank ($N=20$, $X_L = X_b + 3s$, where X_L is the detection limit and X_b is the blank value) for Cu^{2+} and Zn^{2+} ions were found to be 1.8 and $1.5 \mu\text{g} \cdot \text{L}^{-1}$, respectively. The precisions of the determination of Cu^{2+} and Zn^{2+} ions were evaluated by the model solutions containing $50 \mu\text{g} \cdot \text{L}^{-1}$ of ions under the optimum conditions mentioned above. For this purpose, the procedure was repeated 8 times for ions. The sample volume was maintained at 250 mL. The relative standard deviation of the method was found to be below 5% (Table 4). The empirical limit of quantification (LOQ) is the lowest level of analyte that can be accurately and precisely measured, which was found to be $5 \mu\text{g} \cdot \text{L}^{-1}$ for these ions. The proposed preconcentration system shows good enrichment factors with reasonable preconcentration time over other preconcentration methods. Consumptive index (CI) is another efficient way to evaluate the performance of preconcentration system, which expressed by volume of analyte solution (V , mL) and the experimental preconcentration factor (EPF) ratio ($\text{CI} = V/\text{EPF}$), thus indicating the necessary volume to obtain one unit of enrichment factor. The detection limit for proposed method is comparable to those given by many methods; in addition, enrichment factor can be improved by using larger sample volumes. It is important to emphasize that CI values obtained in the present study are lower than those of the most procedures. The experimental preconcentration factors calculated as the ratio of the slope of the calibration graph with and without preconcentration were 38 and 32 for Zn^{2+} and Cu^{2+} ions, while the preconcentration factor as the volume of initial solution (1250 mL) to the volume of eluting solution (8 mL) for all ions was 188.

Table 4 Specification of presented method at optimum conditions for each element

| Parameter | Cu | Zn |
|---|-----------|----------|
| Linear range/ $(\mu\text{g} \cdot \text{mL}^{-1})$ | 0.01–0.41 | 0.01–0.3 |
| Detection limit/ $(\text{ng} \cdot \text{mL}^{-1})$ | 1.8 | 1.5 |
| Loading capacity/ $(\text{mg} \cdot \text{g}^{-1})$ | 13.6 | 13.5 |
| RSD/% | 1.1 | 1.3 |
| Recovery/% | 94.0 | 94.6 |

Application to environmental samples

We have explored the feasibility of the methodology as given in Section “Experiment” using preconcentra-

tion with SDS coated alumina for the determination of Cu^{2+} and Zn^{2+} ions in different environmental matrices by a standard addition method. Reliabilities were checked by spiking experiments and independent analysis. The results for this study are presented in Table 5. The recovery of spiked samples is satisfactorily reason-

Table 5 Recovery of analytes from spiked samples by using proposed method ($N=3$)

| Ion | Added | Found | RSD% | Recovery% |
|------------------|-------|-------|------|-----------|
| Lottus | | | | |
| Cu | 0.0 | 0.102 | 3.6 | — |
| | 0.2 | 0.309 | 2.7 | 103.5 |
| Zn | 0.0 | 0.137 | 3.4 | — |
| | 0.2 | 0.331 | 2.8 | 97.0 |
| Radish | | | | |
| Cu | 0.0 | 0.036 | 4.0 | — |
| | 0.2 | 0.241 | 3.3 | 102.5 |
| Zn | 0.0 | 0.109 | 3.9 | — |
| | 0.2 | 0.313 | 3.2 | 102.0 |
| Eukalptos | | | | |
| Cu | 0.0 | 0.08 | 3.8 | — |
| | 0.2 | 0.275 | 3.0 | 97.5 |
| Zn | 0.0 | 0.973 | 3.6 | — |
| | 0.2 | 1.173 | 2.8 | 99.0 |
| Borage (cowslip) | | | | |
| Cu | 0.0 | 0.04 | 3.9 | — |
| | 0.2 | 0.238 | 3.0 | 99.0 |
| Zn | 0.0 | 0.145 | 3.3 | — |
| | 0.2 | 0.349 | 2.7 | 102.0 |
| Menthe sativa | | | | |
| Cu | 0.0 | 0.086 | 4.1 | — |
| | 0.2 | 0.280 | 3.3 | 97.0 |
| Zn | 0.0 | 0.109 | 3.6 | — |
| | 0.2 | 0.311 | 2.8 | 101.0 |
| Detergent | | | | |
| Cu | 0.0 | 0.007 | 3.9 | — |
| | 0.1 | 0.106 | 3.3 | 99.0 |
| Zn | 0.0 | 1.035 | 3.2 | — |
| | 0.1 | 1.139 | 2.6 | 104.0 |
| Nettle | | | | |
| Cu | 0.0 | 0.276 | 3.0 | — |
| | 0.2 | 0.483 | 2.3 | 103.5 |
| Zn | 0.0 | 0.394 | 3.0 | — |
| | 0.2 | 0.599 | 2.4 | 102.5 |
| Nice | | | | |
| Cu | 0.0 | 0.145 | 3.6 | — |
| | 0.2 | 0.340 | 2.7 | 97.5 |
| Zn | 0.0 | 0.368 | 3.2 | — |
| | 0.2 | 0.562 | 2.8 | 97.0 |
| Cocumert | | | | |
| Cu | 0.0 | 0.098 | 3.9 | — |
| | 0.2 | 0.303 | 3.1 | 102.5 |
| Zn | 0.0 | 0.162 | 3.3 | — |
| | 0.2 | 0.366 | 2.9 | 102.0 |

able and was confirmed using an addition method, which indicates the capability of the system in the determination of Cu^{2+} and Zn^{2+} ions. A good agreement was obtained between the added and measured analyte amounts. The recovery values calculated for the added standards were always higher than 95%, thus confirming the accuracy of the procedure and its independence of the matrix effects.

Conclusion

In the present study, a new solid phase extraction technique was developed based on the preconcentration of Cu^{2+} and Zn^{2+} ions in environmental samples on SDS coated alumina prior to the determination by FAAS. Under conditions given in this article, the determination of Cu^{2+} and Zn^{2+} ions yielded quantitative recoveries. Conditions for quantitative and reproducible preconcentration, elution and FAAS determinations were studied. The method is simple, accurate, and economical and can be applied to the determination of these ions in environmental samples. The system showed reproducibility and reliability in analytical data, with an RSD value of lower than 5% on triplicate experiment. 1.0 g of SDS coated alumina could be used for 10 experiments without any loss in its sorption behavior. The system was successful in preconcentration of analytes from a large sample volume (1800 mL).

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