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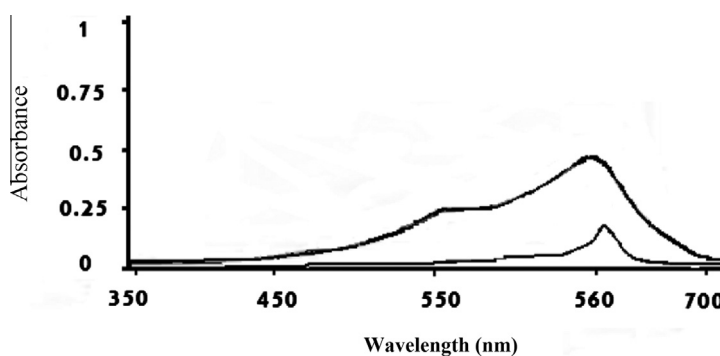
Preconcentration of Sn (II) using the methylene blue on the activated carbon and its determination by spectrophotometry method

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HIGHLIGHTS

- It is a simple spectrophotometric, very sensitivity and low cost method.
- The procedure is based on complex formation between MB and Sn (II).
- The important analytical parameters were investigated.
- The procedure has been successfully applied to determine Sn (II) in soil samples.

GRAPHICAL ABSTRACT



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ABSTRACT

A simple and accurate spectrophotometric method for determination of trace amounts of Sn (II) ion in soil sample was developed by using the methylene blue (MB) in the presence of activated carbon (AC) as the adsorbent Solid Phase Extraction (SPE) of Sn (II) and then determined by UV–Vis. The Beer's law is obeyed over the concentration range of 1–80 ng mL⁻¹ of Sn (II) with the detection limits of 0.34 ng mL⁻¹. The influence of type and volume of eluent, concentration of MB, pH, and amount of AC on sensitivity of spectrophotometric method were optimized. The method has been successfully applied for Sn (II) ion determination in soil sample.

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Introduction

The metal contamination in food has raised public and scientific interest due to their dangerous effects on human health [1–3]. This has led researchers all over the world to study the pollution with heavy metals in air, water, and foods to avoid their harmful effects [4–6]. Tin (Sn) is a toxic metal, which could gather in a human's body and the tissue of animals [7]. The presence of Sn in fresh food of both vegetable and animal origin is highly dependent on the

concentration of Sn in the soil of the area in which the food is produced. The major dietary sources of Sn are fruit products and canned vegetables. Stannous chloride, SnCl₂, is a permitted food additive (E512). Sn is present in some multivitamin and mineral food supplements at levels of up to 0.01 mg in the daily dose recommended by the manufacturer. The tissues and organs that accumulate the highest concentrations of Sn are bone, lymph nodes, liver, lung, ovary, testis and kidney. Reports of acute poisoning have been associated with high concentrations of Sn in drinks or solid foods. However, there is little consistency in the reports in terms of the nature of the foodstuff, or the concentration and chemical form of the Sn, all of which may influence human. Several

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methods including atomic absorption spectrometry [8,9], atomic emission spectrometry [10,11], spectrophotometry [12,13], electrochemical methods [14,15], spectrofluorimetry [16,17] have been developed for the determination of Sn.

Solid Phase Extraction (SPE) has commonly been used as a technique for preconcentration/separation of various inorganic and organic species. SPE is used to enhance the selectivity and sensitivity of the method as it allows for discriminatory binding of analyte to a solid support where it will be accumulated and subsequently eluted with a small volume of solvent. This technique has advantages of higher enrichment factor, absence of emulsion, safety with respect to hazardous samples, minimal costs due to low consumption of reagent, environment friendly, flexibility and easier incorporation into automated analytical techniques [18,19]. Selectivity of the solid phase sorbent towards an analyte depends on the structure of the immobilized organic ligands. Activated carbon (AC) has been widely used for many purposes both in laboratorial and industrial settings. This is due to its ability to absorb both organic and inorganic compounds. Since its introduction in analytical chemistry, enrichment of trace metals using modified AC has been favorably performed with a very high concentration factor in different matrices [20–23]. The sorption of metal ions on activated carbon can be improved in the presence of chelating or precipitating agent. In general, the most successful solid-phase extractors for trace metal ions are those immobilized basically, sulfur and nitrogen containing compounds [21].

In this paper, at first we study the color reaction of methylene blue ($C_{16}H_{18}ClN_3S$) (MB) with Sn (II) and then solid phase extraction of the colored complex with activated carbon (AC) cartridge was investigated. Based on this idea, a highly sensitive, selective and rapid method for the determination of Sn (II) in soil and water was developed.

Experimental

Chemical and reagents

Methylene blue (MB), $SnCl_2 \cdot 2H_2O$, ethanol (96%), HCl (37%) and AC were purchased from Merck (Darmstadt, Germany). All chemicals used were of analytical grade unless otherwise stated. All of the solutions were prepared with distilled water. A stock $6.25 \times 10^{-4} \text{ mol L}^{-1}$ solution of MB was prepared by dissolving 0.02 g of MB in 2 drop HCl (37%) and brought to 100 mL in measuring flask distilled water. Standard stock solution containing 1000 mg L^{-1} of $SnCl_2 \cdot 2H_2O$ was prepared by dissolving 0.1901 g of $SnCl_2 \cdot 2H_2O$ (Merck, Germany) in 1 mL HCl (37%) and brought to 100 mL of distilled water. Working solutions were prepared by appropriate dilution of the standard solution.

Apparatus

ATG 90 UV–Vis spectrophotometer with a 10 mm quartz cell was used for all spectral measurements. All pH measurements were made using pH meter Model 691 Metrohm with a combined glass-calomel electrode. A Buchner funnel connected to vacuum pump (AUTOSCIENCE model) was used. Before use, all glassware was washed with HNO_3 (0.001 mol L^{-1}) and double distillate water.

Preconcentration procedure

In order to SPE for preconcentration of Sn (II), a 250 mL aqueous sample containing of its ions ($10\text{--}30 \text{ ng mL}^{-1}$) and 0.06 mol L^{-1} HCl was mixed with 3.125×10^{-6} MB and then passed at 16.0 mL min^{-1} through the column which containing 100 mg of AC. The adsorbed complex (between Sn (II) and MB) eluted with

2 mL ethanol (96%) with a flow rate of 2.0 mL min^{-1} . The eluent complexes were determined by UV–Vis spectrophotometer. This procedure was carried out for blank sample and difference in absorbance of these samples was used as criteria for determination of Sn (II) concentrations.

Results and discussion

Due to the existence of a donating atom as well as sulfur (S), nitrogen (N) and NH groups in structure of MB, we expect to form complex between MB and Sn (II) and increase the stability and selectivity of its complex. In this research the influences of the analytical parameters including pH, solvents type, amount of adsorbent (AC), elute and sample flow rate and interference ions were investigated.

Type and concentration of acid

In order to choose the most effective adsorbing of the Sn (II) on the sorbent surface, a series of different acids with various concentrations were used. For this purpose 250 mL of sample including $3.125 \times 10^{-6} \text{ mol L}^{-1}$ MB, 30 ng mL^{-1} of Sn (II) with various acids were passed the 0.1 g of AC at 16 mL min^{-1} . Then the retained complexes on the column of AC were eluted with suitable eluent. The results (Fig. 1) showed that the recovery is best when 0.06 mol L^{-1} HCl solution was used. This fact indicates that in acidic media, the positive points of the AC's surface increased and caused the adsorption process of complex increased.

Eluent types and eluent volume

In order to choose the most effective eluent for desorbing the complex of Sn (II) from the sorbent surface, a series of eluent (including ethanol, acetonitrile and acetone) at $1\text{--}5 \text{ mL min}^{-1}$ flow rate were used. For this purpose 250 mL of sample including 0.06 mol L^{-1} HCl, $3.125 \times 10^{-6} \text{ mol L}^{-1}$ of MB and 30 ng mL^{-1} of Sn (II) were passed the 0.1 g of AC at 16 mL min^{-1} . Then the retained complexes on the column of AC were eluted with mention eluent and obtained results are shown in Table 1. Quantitative recoveries of Sn (II) were obtained with 2 mL of ethanol 96% as compared to other eluent; therefore, ethanol was used in the subsequent experiments.

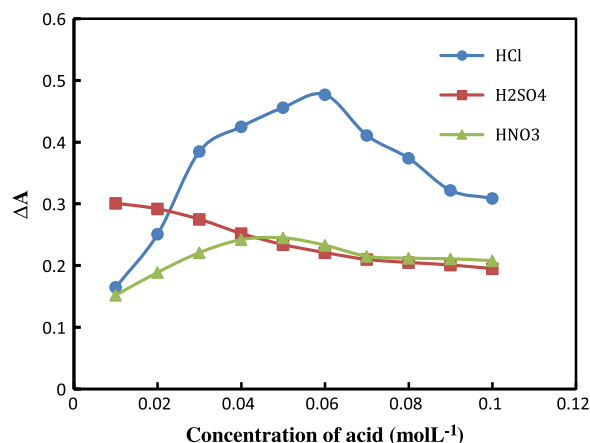


Fig. 1. Effect of type and concentration of acid on the absorbance of complex between Sn (II) and MB.

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