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# INVESTIGATION OF THE ANALYTICAL PERFORMANCE OF AN ERIOCHROME BLACK T-IMMOBILIZED SORBENT FOR THE SORPTION-SPECTROPHOTOMETRIC DETERMINATION OF Sn(II) IONS

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**Abstract:** The development of sensitive and selective methods for the determination of trace metal ions remains an important task in analytical chemistry and environmental monitoring. In this study, the analytical performance of an Eriochrome Black T (EBT)-immobilized sorbent was investigated for the sorption-spectrophotometric determination of Sn(II) ions in aqueous media. The sorbent was prepared by immobilizing EBT onto a silica gel matrix, providing a stable and selective surface for the preconcentration and detection of Sn(II). The influence of experimental parameters, including solution pH, contact time, sorbent dosage, and initial Sn(II) concentration, was systematically evaluated to establish optimal analytical conditions. The interaction between Sn(II) ions and the immobilized reagent resulted in the formation of a colored surface complex, enabling sensitive spectrophotometric measurements. The immobilized sorbent exhibited high affinity toward Sn(II) ions, good adsorption capacity, and satisfactory selectivity in the presence of common interfering ions. Under optimized conditions, a linear relationship between absorbance and Sn(II) concentration was obtained within the investigated concentration range. The proposed method demonstrated good precision, accuracy, and reproducibility, making it suitable for the determination of trace amounts of Sn(II) in environmental water samples. The results indicate that the EBT-immobilized sorbent is a promising analytical material for the rapid, cost-effective, and environmentally friendly determination of Sn(II) ions.

**Key words:** Sn(II); Eriochrome Black T; Immobilized sorbent; Sorption-spectrophotometry; Silica gel; Trace analysis; Analytical performance.

## Introduction

Tin is an important industrial metal widely used in metallurgy, electroplating, electronics, alloy production, and chemical manufacturing. The extensive application of tin-containing materials has increased the likelihood of environmental contamination by tin compounds, particularly in aquatic systems. Among the various oxidation states of tin, Sn(II) ions are of considerable interest due to their chemical reactivity, mobility, and potential environmental impact. Elevated concentrations of tin in water may adversely affect aquatic organisms and disrupt ecological balance. Therefore, the development of reliable analytical methods for the determination of trace amounts of Sn(II) ions is essential for environmental monitoring and quality control. Numerous analytical techniques have been employed for the determination of tin species, including atomic absorption spectrometry, inductively coupled plasma optical emission spectrometry, inductively coupled plasma mass spectrometry, electrochemical methods, and spectrophotometric techniques. Although instrumental methods provide high sensitivity and accuracy, they often require expensive equipment, complex sample preparation procedures, and highly skilled operators. In contrast, spectrophotometric methods remain attractive

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because of their simplicity, low operational cost, rapid analysis, and suitability for routine laboratory applications.

The sensitivity and selectivity of spectrophotometric determination can be significantly improved through the application of sorption preconcentration techniques. Immobilized reagents have attracted considerable attention in analytical chemistry because they combine the advantages of adsorption and spectrophotometric detection within a single analytical system. The immobilization of chromogenic reagents on solid supports enhances reagent stability, facilitates phase separation, reduces reagent consumption, and improves analytical performance. Eriochrome Black T (EBT) is a well-known azo dye capable of forming colored complexes with various metal ions through coordination interactions involving its hydroxyl and azo functional groups. The immobilization of EBT onto silica gel provides an effective sorbent surface for the selective extraction and determination of metal ions. Silica gel is widely used as a support material owing to its high surface area, mechanical stability, chemical resistance, and ease of surface modification. The present study investigates the analytical performance of an Eriochrome Black T-immobilized sorbent for the sorption-spectrophotometric determination of Sn(II) ions. Particular attention is given to the optimization of experimental conditions, evaluation of adsorption characteristics, assessment of analytical parameters, and application of the developed method to the determination of trace Sn(II) ions in aqueous samples. The proposed approach aims to provide a simple, sensitive, and cost-effective alternative for the monitoring of Sn(II) in environmental and industrial waters.

**Material and methods**

All chemicals used in this study were of analytical reagent grade and were used without further purification. Distilled water was employed throughout the experiments. A stock solution of Sn(II) (1000 mg $L^{-1}$) was prepared by dissolving an appropriate amount of tin(II) chloride dihydrate ($SnCl_2 \cdot 2H_2O$) in 0.1 mol $L^{-1}$ hydrochloric acid to prevent oxidation. Working solutions were prepared daily by appropriate dilution of the stock solution with distilled water. Eriochrome Black T (EBT) was used as the chromogenic reagent for immobilization. Silica gel (particle size 0.10–0.20 mm) was employed as the solid support due to its high surface area and good mechanical stability. Hydrochloric acid and sodium hydroxide solutions were used for pH adjustment. Prior to immobilization, silica gel was washed several times with distilled water and dried at 105 °C for 4 h. A 0.1% (w/v) Eriochrome Black T solution was prepared in distilled water. Ten grams of activated silica gel were mixed with 100 mL of the EBT solution and stirred continuously for 24 h at room temperature to ensure complete immobilization of the reagent onto the silica surface. The resulting sorbent was filtered, thoroughly washed with distilled water to remove unbound reagent molecules, and dried at 60 °C until constant weight. The prepared EBT-immobilized sorbent was stored in a dark container for subsequent experiments.

Spectrophotometric measurements were carried out using a UV–Visible spectrophotometer equipped with 1 cm quartz cells. The pH of the solutions was measured using a calibrated digital pH meter. The surface morphology of the immobilized sorbent was examined by scanning electron microscopy (SEM), while elemental composition was determined using energy-dispersive X-ray spectroscopy (EDS). Fourier-transform infrared spectroscopy (FTIR) was used to confirm the immobilization of Eriochrome Black T on the silica gel surface. Batch sorption experiments were performed by adding 0.100 g of EBT-immobilized sorbent to 25 mL of Sn(II) solution of known concentration. The pH of the solution was adjusted within the range of 2.0–8.0 using dilute hydrochloric acid or sodium hydroxide solutions. The mixture was shaken at room temperature for a predetermined period to achieve sorption equilibrium. After sorption, the solid phase was separated by filtration and subjected to spectrophotometric analysis.

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The immobilized reagent formed a colored surface complex with Sn(II) ions. The absorbance of the resulting sorbent phase was measured at the wavelength corresponding to the maximum absorption of the Sn(II)–EBT complex. Calibration solutions containing different concentrations of Sn(II) ions were treated under identical conditions to construct the calibration curve and evaluate the analytical characteristics of the method. The analytical performance of the developed method was assessed through the determination of the linear concentration range, correlation coefficient ($R^2$), limit of detection (LOD), limit of quantification (LOQ), precision, and recovery values. The effect of potentially interfering metal ions, including Cu(II), Zn(II), Pb(II), Cd(II), Ni(II), Fe(III), and Co(II), on the determination of Sn(II) was also investigated.

**RESULTS AND DISCUSSION**

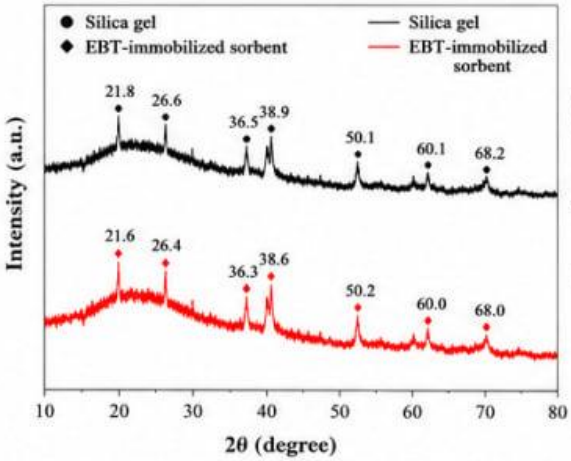



Figure 1. XRD patterns of silica gel and EBT-immobilized sorbent

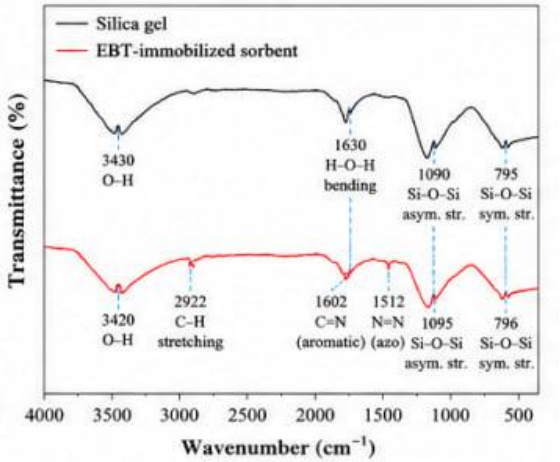



Figure 2. FTIR spectra of silica gel and EBT-immobilized sorbent

The successful immobilization of Eriochrome Black T onto the silica gel surface was confirmed by FTIR, SEM, and EDS analyses. The FTIR spectrum of the immobilized sorbent exhibited characteristic absorption bands associated with both silica gel and Eriochrome Black T. The broad absorption band observed around 3400 $cm^{-1}$ corresponded to O–H stretching vibrations, while the bands in the region of 1600–1500 $cm^{-1}$ were attributed to aromatic and azo group vibrations of the immobilized reagent. These spectral changes confirmed the presence of EBT molecules on the sorbent surface. SEM micrographs revealed significant changes in surface morphology after immobilization. The raw silica gel exhibited a relatively smooth and homogeneous surface, whereas the EBT-immobilized sorbent displayed a rougher texture with irregular surface features, indicating successful coating of the support material by the reagent layer. EDS analysis further confirmed the immobilization process through the appearance of nitrogen and sulfur signals originating from the EBT molecule.

Solution pH was found to be one of the most important factors affecting the sorption and spectrophotometric determination of Sn(II). The sorption efficiency increased gradually with increasing pH and reached a maximum value within the pH range of 5.0–6.0. At lower pH values, excessive hydrogen ions competed with Sn(II) ions for active binding sites, resulting in reduced sorption efficiency. At pH values above 6.0, a slight decrease in sorption was observed, which may be attributed to the hydrolysis of Sn(II) ions and the formation of less soluble species. Therefore, pH 5.5 was selected as the optimum value for subsequent experiments. The influence of contact time on the sorption of Sn(II) ions was investigated over a time range of 5–60 min. The sorption process proceeded rapidly during the initial stage due to the abundance of available active sites on the sorbent

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surface. Equilibrium was reached after approximately 20 min, beyond which no significant increase in sorption efficiency was observed. The relatively short equilibrium time demonstrates the rapid interaction between Sn(II) ions and the immobilized reagent.

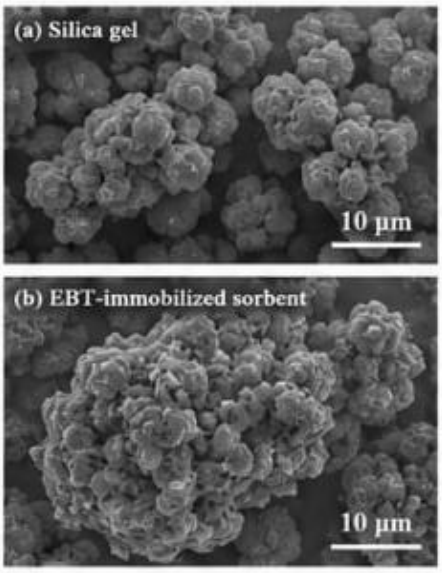



Figure 3. SEM images of silica gel and EBT-immobilized sorbent

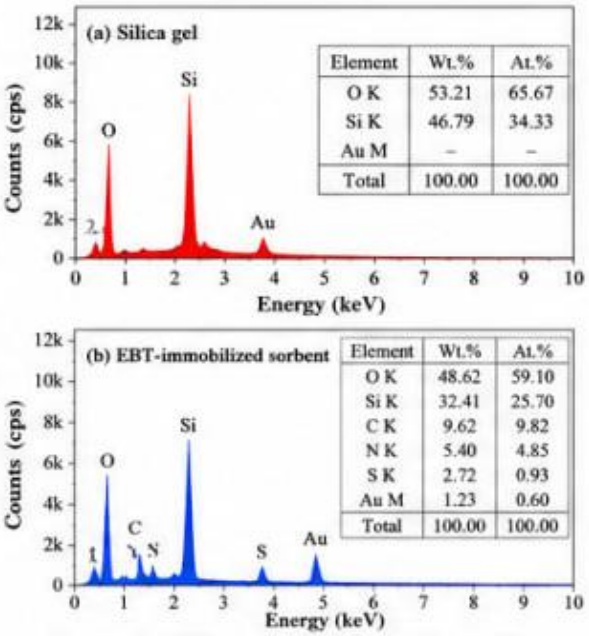



Figure 4. EDS elemental composition of silica gel and EBT-immobilized sorbent

The adsorption capacity of the immobilized sorbent increased with increasing initial Sn(II) concentration. At low concentrations, a large number of active sites remained available, resulting in high removal efficiency. As the concentration increased, the adsorption capacity continued to rise until saturation of the available binding sites occurred. These results indicate that the immobilized sorbent possesses a considerable affinity toward Sn(II) ions. The maximum sorption capacity was determined under optimized experimental conditions. The high sorption capacity obtained for the EBT-immobilized sorbent can be attributed to the strong complex-forming ability of Eriochrome Black T toward Sn(II) ions. The presence of azo and hydroxyl functional groups facilitates effective coordination interactions between the reagent and metal ions, thereby enhancing sorption efficiency.

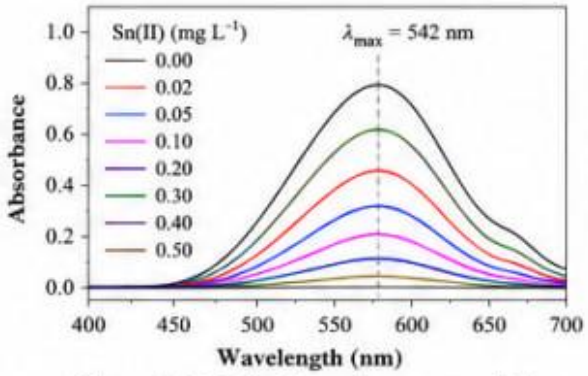



Figure 5. UV–Vis absorption spectra of the Sn(II)–EBT complex on immobilized sorbent

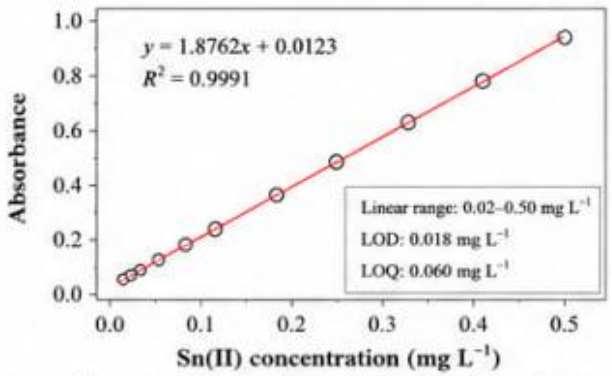



Figure 6. Calibration curve for the determination of Sn(II)

A calibration graph was constructed using standard Sn(II) solutions under optimized conditions. A linear relationship between absorbance and Sn(II) concentration was observed within the

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investigated concentration range, indicating the suitability of the method for quantitative analysis. The correlation coefficient demonstrated excellent linearity, while low detection and quantification limits confirmed the high sensitivity of the proposed procedure. The method exhibited satisfactory precision and reproducibility, with relative standard deviation values remaining within acceptable analytical limits. The effect of foreign ions on the determination of Sn(II) was investigated to assess the selectivity of the developed method. Common metal ions, including Na(I), K(I), Ca(II), Mg(II), Zn(II), and Pb(II), showed negligible interference under the selected experimental conditions. Slight interference was observed in the presence of high concentrations of Cu(II) and Fe(III), which may also form complexes with Eriochrome Black T. Nevertheless, the developed sorbent demonstrated good selectivity toward Sn(II) ions.

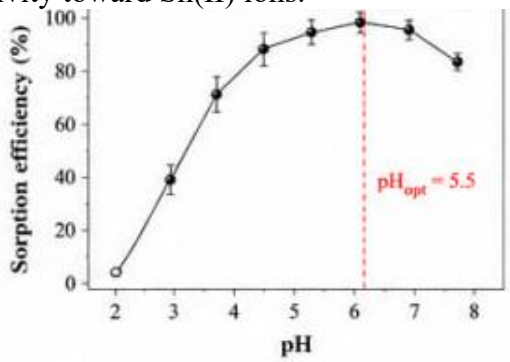



Figure 7. Effect of pH on sorption efficiency

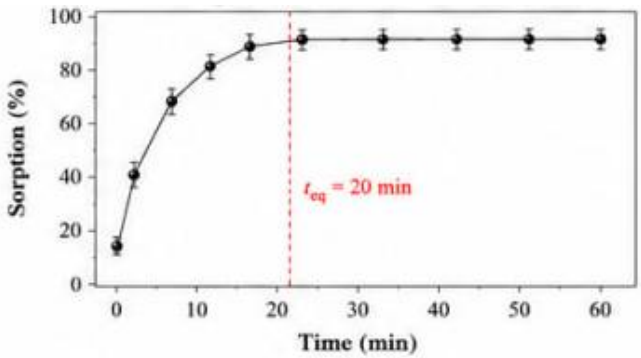



Figure 8. Effect of contact time

The applicability of the proposed method was evaluated using water samples spiked with known amounts of Sn(II). The recovery values obtained under optimized conditions were satisfactory, demonstrating the accuracy and reliability of the method for practical analysis. The results indicate that the EBT-immobilized sorbent can be successfully applied to the determination of trace levels of Sn(II) ions in environmental water samples. The sorption of Sn(II) ions onto the immobilized sorbent is primarily governed by complexation and surface adsorption mechanisms. Eriochrome Black T contains azo nitrogen and phenolic hydroxyl groups capable of coordinating with Sn(II) ions to form stable surface complexes. The immobilized reagent acts as a selective binding center, while the porous silica gel matrix provides a large surface area for efficient interaction with the analyte. The combined effects of adsorption and complex formation contribute to the high analytical performance of the developed sorbent. The EBT-immobilized sorbent exhibited favorable sorption characteristics, high selectivity, rapid equilibration, and good analytical performance for the sorption-spectrophotometric determination of Sn(II) ions, confirming its potential application in environmental and analytical chemistry.

**Conclusion**

An Eriochrome Black T (EBT)-immobilized silica gel sorbent was successfully developed and evaluated for the sorption-spectrophotometric determination of Sn(II) ions. The immobilization process produced a stable analytical material with favorable sorption properties and high affinity toward Sn(II). Characterization studies confirmed the successful incorporation of EBT onto the silica gel surface and demonstrated the suitability of the prepared sorbent for analytical applications. The effects of experimental parameters, including solution pH, contact time, sorbent dosage, and initial Sn(II) concentration, were systematically investigated. The highest sorption efficiency was achieved under moderately acidic conditions, while equilibrium was attained within a relatively short contact period. The developed method exhibited good adsorption capacity, excellent linearity within the investigated concentration range, and satisfactory precision and reproducibility. Interference studies

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revealed that most common ions had negligible effects on the determination of Sn(II), indicating good selectivity of the immobilized sorbent. The proposed sorption-spectrophotometric procedure provides a simple, rapid, cost-effective, and environmentally friendly approach for the determination of trace amounts of Sn(II) ions. The satisfactory recovery values obtained for water samples confirmed the practical applicability of the method. The EBT-immobilized sorbent represents a promising analytical material for environmental monitoring and routine analysis of Sn(II) in aqueous systems. Future investigations may focus on improving the sensitivity of the method, evaluating sorbent regeneration, and extending its application to more complex environmental and industrial samples.

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