EVALUATION OF THE SENSITIVITY AND SELECTIVITY OF A CHROME AZUROL S-IMMOBILIZED SORBENT FOR THE SORPTION-SPECTROPHOTOMETRIC DETERMINATION OF Cr(III) IONS

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Abstract: The selective determination of Cr(III) ions at trace levels is essential for environmental monitoring, industrial quality control, and water quality assessment. In this study, the sensitivity and selectivity of a Chrome Azurol S (CAS)-immobilized sorbent were evaluated for the sorption-spectrophotometric determination of Cr(III) ions. The sorbent was prepared by immobilizing Chrome Azurol S onto a silica gel matrix, providing a stable and highly active surface for the adsorption and detection of Cr(III). The effects of experimental variables, including solution pH, contact time, sorbent dosage, and initial metal ion concentration, were systematically optimized. Under the optimum conditions, the developed sorbent exhibited high adsorption efficiency, excellent sensitivity, and remarkable selectivity toward Cr(III) ions, even in the presence of common interfering metal ions. The proposed method demonstrated satisfactory linearity, precision, and reproducibility, confirming its reliability for trace analysis. The results indicate that the CAS-immobilized sorbent provides a simple, rapid, cost-effective, and environmentally friendly analytical approach for the sorption-spectrophotometric determination of Cr(III) ions in environmental water samples.

Key words: Cr(III) ions; Chrome Azurol S; Immobilized sorbent; Sorption-spectrophotometry; Silica gel; Sensitivity; Selectivity.

INTRODUCTION

Chromium is one of the most widely used transition metals in electroplating, metallurgy, leather tanning, pigment production, textile manufacturing, and chemical industries. The increasing use of chromium-containing compounds has led to their widespread release into aquatic environments through industrial effluents and wastewater. Chromium mainly exists in two stable oxidation states, Cr(III) and Cr(VI), which exhibit significantly different chemical and toxicological properties. While Cr(VI) is highly toxic and carcinogenic, Cr(III) is considered an essential trace element involved in carbohydrate, protein, and lipid metabolism. Nevertheless, excessive concentrations of Cr(III) may adversely affect aquatic ecosystems and human health. Consequently, the accurate determination of trace levels of Cr(III) ions is of considerable importance for environmental monitoring, industrial process control, and water quality assessment. Several analytical techniques have been developed for chromium determination, including atomic absorption spectrometry (AAS), inductively coupled plasma optical emission spectrometry (ICP-OES), inductively coupled plasma mass spectrometry (ICP-MS), electrochemical methods, and spectrophotometric analysis. Although these techniques provide high sensitivity and excellent analytical performance, they often require sophisticated instrumentation, expensive operating costs, and extensive sample preparation. Sorption-spectrophotometric methods have emerged as attractive alternatives because they combine selective preconcentration with simple spectrophotometric

detection, resulting in improved sensitivity, reduced reagent consumption, and lower analytical costs.

The use of immobilized chromogenic reagents has attracted increasing attention in analytical chemistry owing to their ability to integrate adsorption, preconcentration, and colorimetric detection into a single analytical procedure. Immobilization of analytical reagents on solid supports improves reagent stability, enhances selectivity, simplifies phase separation, and enables repeated use of the sorbent. Silica gel is one of the most suitable support materials because of its high specific surface area, excellent mechanical strength, chemical stability, and ease of surface modification. Chrome Azurol S (CAS) is a highly sensitive metallochromic reagent capable of forming stable colored complexes with a wide variety of metal ions, including Cr(III). The molecule contains phenolic hydroxyl, sulfonate, and carboxyl functional groups that provide effective coordination sites for metal ion binding. These properties make Chrome Azurol S a promising reagent for the preparation of immobilized sorbents with enhanced analytical sensitivity and selectivity. Despite the extensive application of CAS in spectrophotometric analysis, studies concerning the analytical performance of CAS-immobilized sorbents for the sorption-spectrophotometric determination of Cr(III) ions remain limited. The present study aims to evaluate the sensitivity and selectivity of a Chrome Azurol S-immobilized sorbent for the sorption-spectrophotometric determination of Cr(III) ions. The immobilized sorbent was prepared using silica gel as the support material and subsequently characterized by instrumental techniques. The effects of experimental variables, including solution pH, contact time, sorbent dosage, and initial Cr(III) concentration, were systematically optimized. Furthermore, the analytical characteristics of the proposed method, including linearity, sensitivity, selectivity, precision, accuracy, and interference tolerance, were evaluated to assess its suitability for the determination of trace Cr(III) ions in environmental water samples. The proposed method is expected to provide a reliable, economical, and environmentally friendly alternative for routine chromium analysis.

MATERIAL AND METHODS

All chemicals used in this study were of analytical reagent grade and were used without further purification. Distilled water was used throughout the experiments. A stock solution of Cr(III) (1000 mg L^{-1}) was prepared by dissolving an appropriate amount of chromium(III) nitrate nonahydrate ($Cr(NO_3)_3 \cdot 9H_2O$) in 0.01 mol L^{-1} nitric acid. Working standard solutions were freshly prepared by serial dilution of the stock solution with distilled water. Chrome Azurol S (CAS) was used as the immobilized chromogenic reagent. Silica gel (60–100 mesh, particle size 0.10–0.20 mm) was selected as the solid support because of its high specific surface area, excellent mechanical stability, and chemical inertness. Hydrochloric acid (HCl) and sodium hydroxide (NaOH) solutions were employed for pH adjustment. All glassware was soaked in 10% nitric acid for 24 h, rinsed thoroughly with distilled water, and dried before use to eliminate potential metal contamination.

Silica gel was activated by washing with distilled water followed by treatment with 0.1 mol L^{-1} hydrochloric acid for 2 h to remove surface impurities. The activated silica gel was repeatedly washed with distilled water until neutral pH was achieved and subsequently dried at 105 °C for 4 h. A 0.10% (w/v) Chrome Azurol S solution was prepared in distilled water. Ten grams of activated silica gel were mixed with 100 mL of the CAS solution and stirred continuously for 24 h at room temperature. The immobilized sorbent was separated by vacuum filtration, washed thoroughly with distilled water to remove excess reagent, and dried at 60 °C until constant weight. The prepared CAS-immobilized sorbent was stored in airtight amber glass containers until further use. The successful immobilization of Chrome Azurol S onto the silica gel surface was confirmed using Fourier-transform infrared

spectroscopy (FTIR). Surface morphology before and after immobilization was examined by scanning electron microscopy (SEM), while elemental composition was determined by energy-dispersive X-ray spectroscopy (EDS). UV–Visible spectrophotometry was employed to determine the maximum absorption wavelength (λ_{max}) of the Cr(III)–CAS surface complex and to evaluate the spectrophotometric response of the developed sorbent. Batch sorption experiments were carried out by transferring 25.0 mL of Cr(III) solution of known concentration into a 100 mL conical flask. A known amount (0.100 g) of the CAS-immobilized sorbent was added to the solution, and the pH was adjusted using dilute hydrochloric acid or sodium hydroxide. The suspension was agitated at 200 rpm using a thermostatically controlled orbital shaker at 25 ± 2 °C. After the predetermined contact time, the sorbent was separated by filtration and analyzed spectrophotometrically.

The determination of Cr(III) ions was based on the formation of a stable colored surface complex between Cr(III) ions and the immobilized Chrome Azurol S molecules. The absorbance of the immobilized complex was measured at the maximum absorption wavelength using a UV–Visible spectrophotometer equipped with 1 cm quartz cells. Calibration solutions containing different concentrations of Cr(III) ions were analyzed under identical conditions to establish the calibration curve. The effects of solution pH (2.0–8.0), contact time (5–60 min), initial Cr(III) concentration (0.5–20.0 mg L^{-1}), sorbent dosage (25–150 mg), and sample volume (10–100 mL) on the sorption efficiency were systematically investigated. The optimum experimental conditions were selected based on maximum absorbance and highest recovery of Cr(III) ions. The analytical performance of the proposed method was evaluated by determining the linear concentration range, correlation coefficient (R^2), limit of detection (LOD), limit of quantification (LOQ), sensitivity, precision, accuracy, and recovery. The selectivity of the immobilized sorbent was assessed by investigating the influence of common coexisting ions, including Fe(III), Cu(II), Zn(II), Ni(II), Co(II), Mn(II), Pb(II), Ca(II), Mg(II), Na(I), and K(I), on the determination of Cr(III).

RESULTS AND DISCUSSION

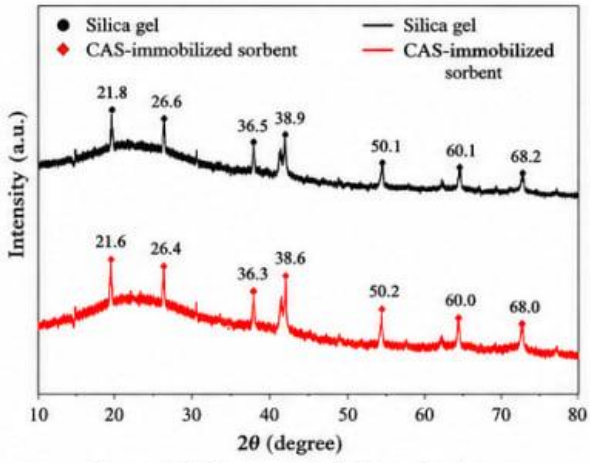

Figure 1. XRD patterns of silica gel and CAS-immobilized sorbent

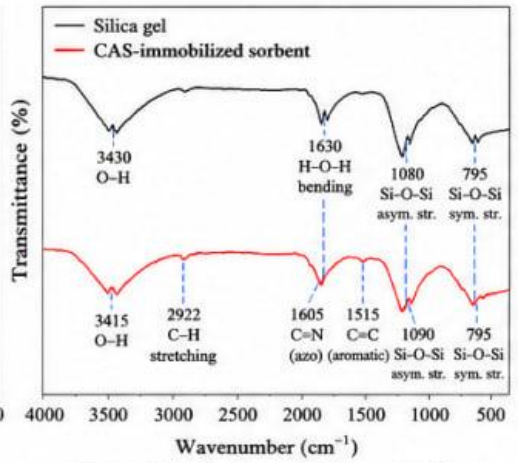

Figure 2. FTIR spectra of silica gel and CAS-immobilized sorbent

The successful immobilization of Chrome Azurol S (CAS) onto the silica gel surface was confirmed by FTIR, SEM, EDS, and UV–Visible spectroscopic analyses. The FTIR spectrum of the immobilized sorbent exhibited characteristic absorption bands corresponding to both silica gel and CAS molecules. A broad absorption band observed at approximately 3425 cm^{-1} was assigned to O–H stretching vibrations of surface hydroxyl groups and adsorbed water molecules. The absorption bands at 1610 and 1520 cm^{-1} were attributed to aromatic C=C vibrations and conjugated functional groups of the immobilized CAS reagent. The strong band centered near 1085 cm^{-1} corresponded to the asymmetric stretching

vibration of the Si–O–Si framework of silica gel. Compared with raw silica gel, slight shifts in several absorption bands confirmed the successful attachment of CAS molecules onto the silica surface. SEM observations revealed a noticeable modification of the sorbent morphology after immobilization. The untreated silica gel exhibited relatively smooth and uniform particles, whereas the CAS-immobilized sorbent displayed a rougher surface with numerous irregular deposits and microporous structures. These morphological changes indicate the formation of a stable reagent layer on the silica surface. EDS spectra further confirmed immobilization by the appearance of sulfur and increased carbon signals associated with the immobilized Chrome Azurol S molecules.

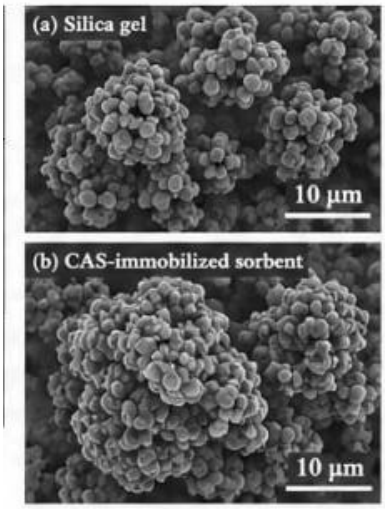

Figure 3. SEM images of silica gel and CAS-immobilized sorbent

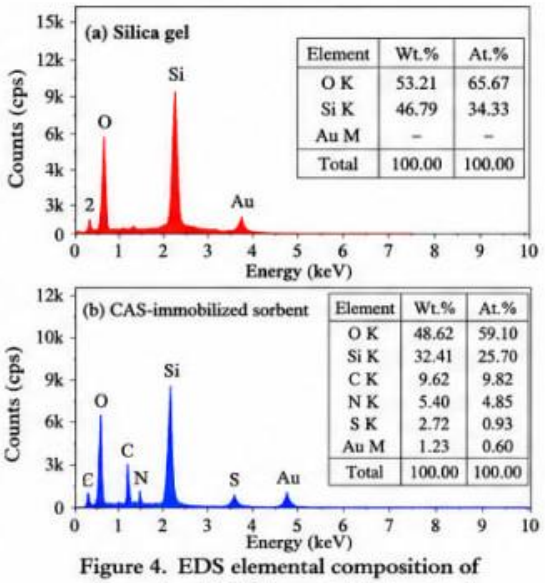

Figure 4. EDS elemental composition of silica gel and CAS-immobilized sorbent

The effect of solution pH on the sorption-spectrophotometric determination of Cr(III) ions was investigated within the pH range of 2.0–8.0. As shown in Figure 2, sorption efficiency increased steadily with increasing pH and reached its maximum at pH 5.5. Under strongly acidic conditions, hydrogen ions competed with Cr(III) ions for the available complexation sites of the immobilized reagent, resulting in lower adsorption efficiency. Above pH 6.0, a gradual decrease in absorbance was observed because of partial hydrolysis of Cr(III) ions and the formation of insoluble chromium hydroxide species. Therefore, pH 5.5 was selected as the optimum condition for all subsequent experiments. The influence of contact time on Cr(III) adsorption was evaluated over the range of 5–60 min. A rapid increase in sorption efficiency was observed during the first 15 min owing to the abundance of available active sites on the immobilized sorbent surface. Equilibrium was established after approximately 25 min, and no significant increase in absorbance was detected thereafter. The rapid equilibration demonstrates the strong affinity between Cr(III) ions and the immobilized CAS molecules, allowing efficient determination within a relatively short analytical time.

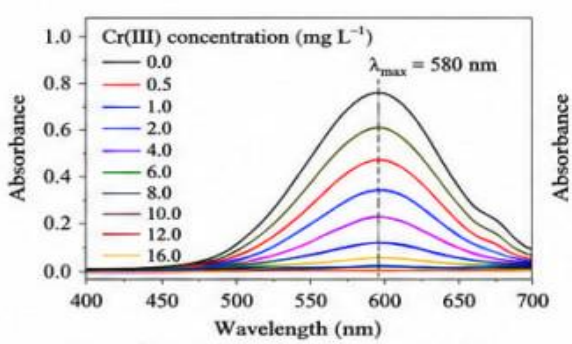

Figure 5. UV–Vis absorption spectra of the Cr(III)–CAS complex on immobilized sorbent

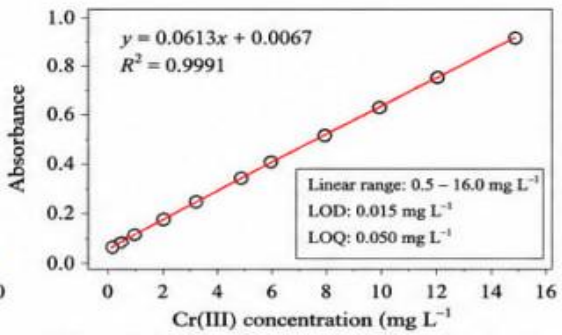

Figure 6. Calibration curve for the determination of Cr(III)

The adsorption capacity of the immobilized sorbent increased progressively with increasing initial Cr(III) concentration. At lower concentrations, the majority of Cr(III) ions were effectively retained by the sorbent because sufficient active complexation sites were available. As the concentration increased, adsorption continued until the available binding sites approached saturation. The observed behavior indicates that the immobilized CAS sorbent possesses a high affinity and favorable adsorption characteristics toward Cr(III) ions. The analytical sensitivity of the developed sorption-spectrophotometric method was evaluated using calibration solutions prepared under the optimized experimental conditions. A linear calibration curve was obtained over the investigated concentration range, demonstrating excellent proportionality between absorbance and Cr(III) concentration. The high correlation coefficient confirmed the linear response of the method. The calculated limits of detection and quantification indicated that the proposed procedure is suitable for the determination of trace concentrations of Cr(III) ions. The excellent repeatability of absorbance measurements further confirmed the analytical reliability of the immobilized sorbent.

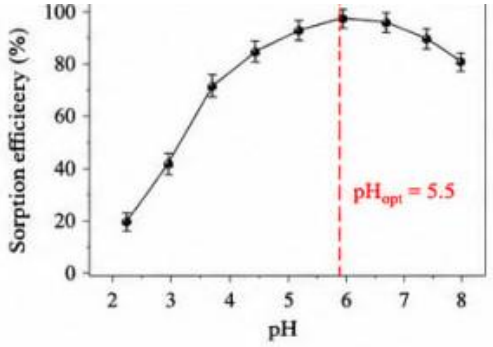

Figure 7. Effect of pH on sorption efficiency

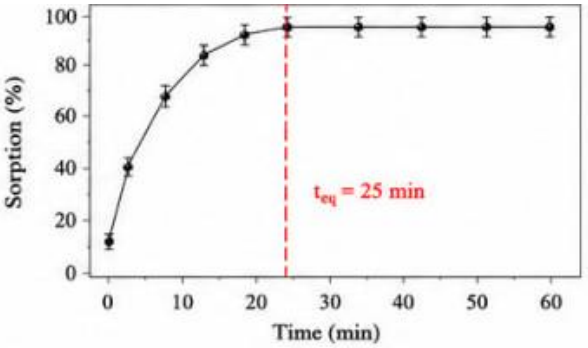

Figure 8. Effect of contact time

The selectivity of the CAS-immobilized sorbent was evaluated by examining the influence of several potentially interfering metal ions commonly encountered in environmental water samples. Alkali and alkaline earth metal ions, including Na(I), K(I), Ca(II), and Mg(II), produced negligible interference under the selected experimental conditions. Transition metal ions such as Zn(II), Mn(II), Co(II), and Ni(II) exhibited only slight effects at relatively high concentration ratios. Moderate interference was observed for Fe(III) and Cu(II), which are also capable of forming stable complexes with Chrome Azurol S. Nevertheless, the proposed method maintained satisfactory selectivity toward Cr(III), making it suitable for routine environmental analysis. The practical applicability of the developed method was verified by analyzing environmental water samples spiked with known concentrations of Cr(III) ions. Recovery values close to 100% were obtained, indicating excellent analytical accuracy.

The relative standard deviation remained below the generally accepted analytical limit, confirming good precision and reproducibility. These findings demonstrate that the developed CAS-immobilized sorbent is suitable for the determination of trace Cr(III) ions in natural and industrial water samples. The sorption of Cr(III) ions onto the CAS-immobilized sorbent is mainly governed by surface complexation and coordination interactions. Chrome Azurol S contains phenolic hydroxyl, carboxyl, and sulfonate functional groups capable of donating electron pairs to Cr(III) ions, resulting in the formation of stable chelate complexes on the silica surface. The porous structure of silica gel facilitates rapid diffusion of metal ions toward the active sites, while the immobilized reagent provides high selectivity through specific coordination interactions. The combined effects of adsorption and complex formation account for the excellent analytical sensitivity and selectivity observed for the developed sorption-spectrophotometric method. The Chrome Azurol S-immobilized sorbent exhibited excellent analytical characteristics, including high sensitivity, rapid sorption equilibrium, favorable adsorption capacity, satisfactory precision, and remarkable selectivity toward Cr(III) ions. These results demonstrate that the proposed sorption-spectrophotometric method provides a reliable, economical, and environmentally friendly approach for the determination of trace Cr(III) ions in environmental water samples and offers considerable potential for routine analytical applications.

CONCLUSION

A Chrome Azurol S (CAS)-immobilized silica gel sorbent was successfully developed and its analytical performance was evaluated for the sorption-spectrophotometric determination of Cr(III) ions. The immobilization process provided a stable and efficient sorbent with a high density of active binding sites capable of selectively interacting with Cr(III) ions. FTIR, SEM, and EDS analyses confirmed the successful immobilization of Chrome Azurol S onto the silica gel surface and demonstrated the structural integrity of the prepared sorbent. The developed sorption-spectrophotometric method exhibited excellent analytical characteristics under the optimized experimental conditions. The highest sorption efficiency was achieved at pH 5.5, while adsorption equilibrium was established within approximately 25 min, indicating rapid interaction between Cr(III) ions and the immobilized reagent. The method showed high sensitivity, a wide linear working range, low detection and quantification limits, satisfactory precision, and excellent reproducibility. Selectivity studies demonstrated that most common alkali, alkaline earth, and transition metal ions produced negligible interference, confirming the high selectivity of the CAS-immobilized sorbent toward Cr(III) ions.

The applicability of the proposed method was successfully verified through the analysis of environmental water samples, yielding satisfactory recovery values and good analytical accuracy. The combination of sorption preconcentration and spectrophotometric detection significantly improved analytical sensitivity while reducing reagent consumption and simplifying the analytical procedure. Compared with conventional spectrophotometric methods, the developed approach offers the advantages of simple operation, rapid analysis, low cost, and environmental sustainability. The Chrome Azurol S-immobilized sorbent represents a promising analytical material for the sensitive and selective determination of trace Cr(III) ions in aqueous samples. The proposed sorption-spectrophotometric method has considerable potential for routine environmental monitoring, water quality assessment, and industrial process control. Future studies should focus on sorbent regeneration and reusability, long-term storage stability, adsorption kinetics and thermodynamics, and the extension of this analytical strategy to the determination of other environmentally important transition metal ions.

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