

ECO-FRIENDLY Fe₃O₄ NANOCOMPOSITES FUNCTIONALIZED WITH CARBOXYMETHYL-β- CYCLODEXTRIN (CM-β-CD): A NOVEL ADSORBENT FOR BORON ELIMINATION

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ABSTRACT

The preparation of CM-β-CD-Fe₃O₄ nanoparticles involved preparing a magnetic core with a functional organic shell. This magnetic nanocomposite was composed of magnetite (Fe₃O₄) nanoparticles functionalized with a carboxymethyl-β-cyclodextrin coating. This hybrid material combines facile magnetic recovery with enhanced adsorption performance, rendering applications in water purification and environmental remediation. This study primarily aimed to develop a nanocomposite and investigate its use in eliminating dyes, such as boron, from wastewater. The synthesized material was characterized to confirm its structural, morphological, and surface properties. Experimental studies demonstrated that 98% of boron was removed under optimal conditions at pH 5, with an adsorbent dosage of 0.3 g and a contact time of 60 minutes. The findings of this study confirm that the developed adsorbent exhibits excellent capability for boron removal under optimized operational parameters. The high removal efficiency achieved under controlled conditions of pH, dosage, concentration, and contact time underscores the robust interaction between the adsorbent surface and boron species. These findings indicate that the material holds considerable promise for application in water purification systems. Moreover, its performance suggests its suitability for scalable environmental treatment processes, thereby justifying further investigation into its regeneration, long-term stability, and practical deployment

Keywords: Magnetic Core, Functional Organic Shell, Adsorbent, Nanocomposite, Surface Properties.

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INTRODUCTION

Boron is a trace element that naturally occurs in surface and groundwater owing to the weathering of rocks and soil. Nonetheless, elevated boron concentrations, primarily resulting from agricultural fertilisers, glass and detergent manufacturing, and mining activities, have emerged as a significant environmental concern. Excessive levels of boron can lead to severe ecological and health impacts, including reproductive toxicity, developmental disorders, and phytotoxic effects in plants at higher concentrations.^{1,2} Consequently, regulatory guidelines restrict boron levels in drinking water to low mg/L ranges, underscoring the urgent need for efficient and reliable removal technologies.² The various treatment methodologies are acknowledged as a cost-effective and operationally straightforward approach with high efficacy across diverse water systems.³ Traditional adsorbents such as activated carbon, zeolites, and layered double hydroxides have been explored for boron removal; however, their practical application is frequently constrained by low selectivity, substantial material requirements, and challenges in regeneration.⁴ In recent years, magnetic nanomaterials, particularly Fe₃O₄ NPs emerged as promising adsorbents due to their extensive surface area and adjustable surface properties.^{5,6} However, unmodified Fe₃O₄ nanoparticles tend to aggregate and lack sufficient functional groups for effective boron binding, thereby diminishing their adsorption performance.⁷ To mitigate these limitations, surface modification with organic or polymeric compounds has been extensively investigated to enhance stability, selectivity, and adsorption efficiency. Cyclodextrins (CDs), a class of cyclic oligosaccharides, exhibit a distinctive architecture characterised by a hydrophobic inner cavity and a hydrophilic outer surface. Their derivatives, such as carboxymethyl-β-cyclodextrin (CM-β-CD), are rich in functional groups that facilitate interactions with various contaminants while enhancing dispersion stability. The incorporation of CM-β-

CD with Fe_3O_4 nanoparticles resulted in a synergistic system that combined magnetic separability with an improved adsorption affinity, rendering it a sustainable option for water purification. Despite these advancements, a significant research gap persists in the application of cyclodextrin-functionalized magnetic nanocomposites specifically for boron removal. The study addresses the gap by synthesizing and characterizing CM- β -CD- Fe_3O_4 nanocomposites as a sustainable and competent adsorbent for boron elimination from aqueous systems. Furthermore, this research aligns with SDG-6, supporting sustainable materials aimed at enhancing water quality and ensuring safe access to clean water.

EXPERIMENTAL

Material and Methods

Boric acid (H_3BO_3) was used to prepare a stock boron solution with a concentration of 1000 mg/L. Carboxymethyl- β -cyclodextrin (CM- β -CD, 99%), ammonium hydroxide (NH_4OH , 98%), iron (III) chloride hexahydrate ($\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$, 99.0%), and iron (II) chloride tetrahydrate ($\text{FeCl}_2 \cdot 4\text{H}_2\text{O}$, 98.0%) were employed for the synthesis of magnetic nanocomposites. Sodium hydroxide (NaOH , 98%), hydrochloric acid (HCl , 37%), and nitric acid (HNO_3 , 69%) were employed for pH adjustment and solution preparation. All reagents were of analytical grade and used as received without additional purification.

Synthesis of Beta-cyclodextrin

The synthesis of CM- β -CD was achieved through alkaline carboxymethylation. Briefly, 1.86 g of NaOH was dissolved in 7.5 mL of double-distilled water, followed by dropwise addition of 5.5 mL of 16.3% chloroacetic acid under constant stirring. The reaction proceeds via the activation of the cyclodextrin hydroxyl groups to form alkoxide ions, followed by a nucleophilic substitution with chloroacetic acid (Fig.-1 and 2). The resulting CM- β -CD was precipitated using cold methanol, isolated, and vacuum-dried at 50 °C. Carboxymethylation occurs through alkaline activation of β -cyclodextrin followed by nucleophilic substitution with chloroacetic acid, leading to the formation of carboxymethyl- β -cyclodextrin.

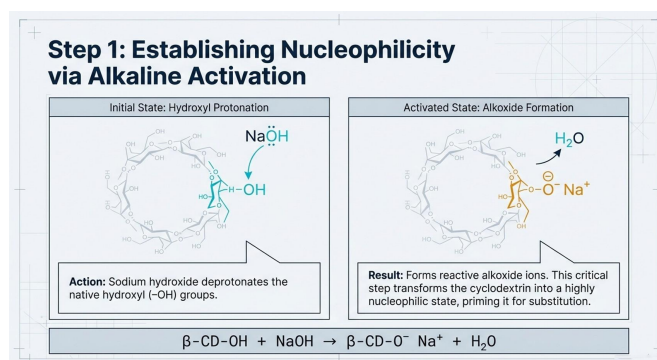


Fig.-1: Activation of β -cyclodextrin

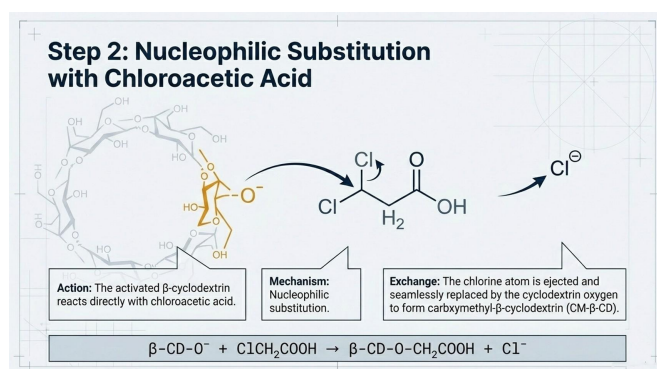


Fig.-2: Carboxymethylation

RESULTS AND DISCUSSION

Physicochemical Evaluation

The molecular structure and the functional groups of Carboxymethyl- β -cyclodextrin functionalized Fe_3O_4 nanoparticles are shown in Fig.-3(a). The spectrum exhibits distinct broad absorption bands below 900 cm^{-1} , attributed to Fe–O vibrations.

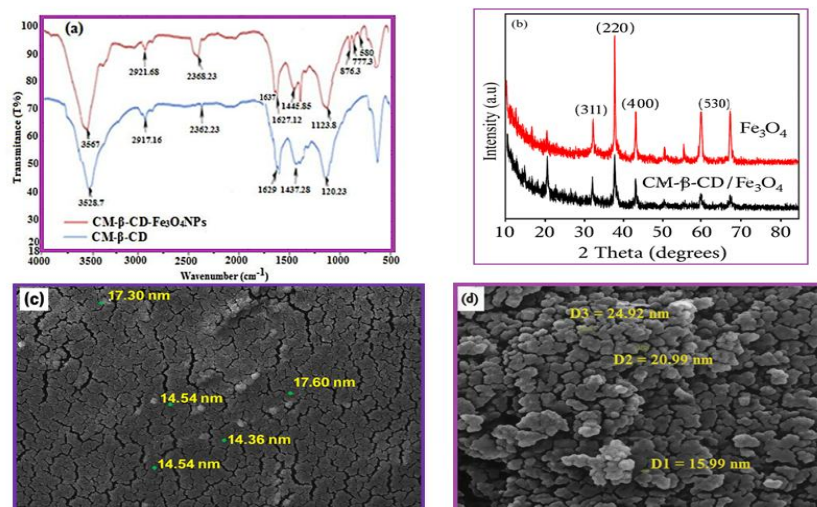


Fig.-3: (a) FT-IR Spectrum; (b) XRD Patterns; (c and d) SEM Images

The XRD pattern of the Carboxymethyl- β -cyclodextrin-functionalized Fe_3O_4 nanoparticles (Fig.-3b). The prominent diffraction peaks observed at 2θ values of approximately 69.17° , 54.72° , and 81.17° are indexed to the (311), (220), and (400) of JCPDS card No. 19-0629 with an estimated size of coherent crystalline of 12.14 nm. Figure-3(c) and 3(d) present SEM images that illustrate the surface morphology and particle size distribution. The calculated particle sizes, based on the Debye–Scherrer formula, ranged from 14 to 25 nm, in good agreement with the crystallite dimensions obtained from XRD data.¹² The point of zero charge (pH_{pzc}) of the CM- β -CD- Fe_3O_4 nanoparticles was found to be 5.5 (Fig.-4). At pH values lower than the pH_{pzc} , the adsorbent surface carries a net positive charge, whereas adjustment of the solution pH to approximately 5 promotes the development of negatively charged surface sites that enhance boron uptake. Under these conditions, boron species interact with the nanocomposite primarily through electrostatic attraction toward these negatively charged functional groups. The solution pH, therefore, plays a critical role in controlling surface charge characteristics, adsorption behaviour, and overall removal efficiency, as illustrated in Fig.-4.

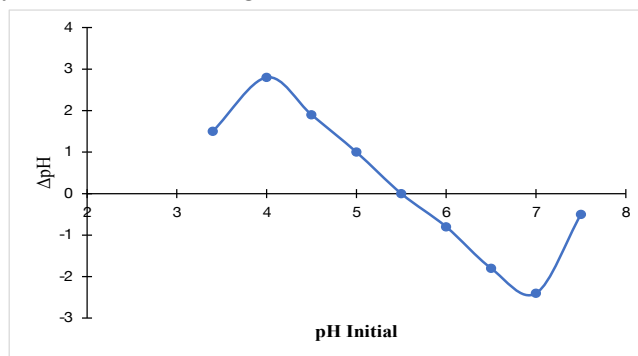


Fig.-4: Impact of Initial pH on Adsorption Performance

Impact of Solution pH on Adsorption Behaviour

The boron adsorption behaviour as a function of solution pH was studied using CM- β -CD functionalized Fe_3O_4 nanoparticles under batch conditions within a pH range of 2.0 to 8.0 (Fig.-5). The adsorption

performance peaked at pH 5.0, which was consequently chosen as the optimal condition for later adsorption investigations. When the solution pH rises above 5.0, the adsorbent surface becomes progressively more negatively charged, which diminishes boron uptake due to enhanced electrostatic repulsion between the negatively charged surface sites and boron species.

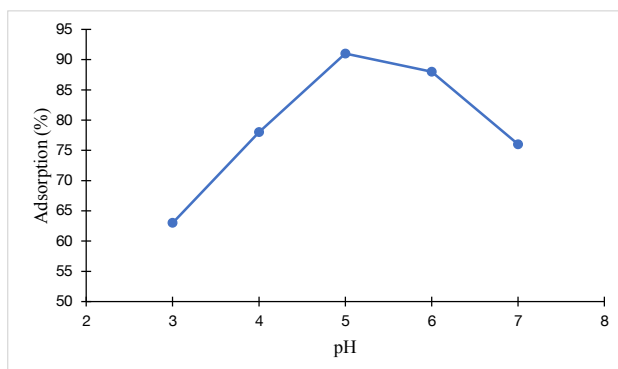


Fig.-5: Plot Adsorption vs pH

When the pH becomes higher than 5, the surface of your adsorbent gains more negative charge. At the same time, boron species in the solution are also negatively charged. Owing to this electrostatic repulsion, boron cannot easily attach to the surface. Therefore, adsorption decreases. For a specific initial adsorbate concentration, the adsorption efficiency is largely governed by the adsorbent dosage.¹³ Its impact was examined through batch adsorption tests using dosages between 0.05 and 1.0 g, as shown in Fig.-6. As shown in the figure, 0.3 g of the adsorbent was identified as the optimal dosage.

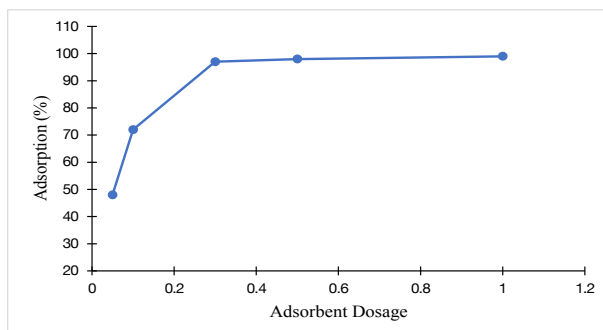


Fig.-6: Influence of Adsorbent Dosage

At dosages higher than the optimum value, a greater number of adsorption sites remain unsaturated, resulting in no significant improvement in adsorption efficiency.¹⁴ Conversely, at low adsorbent/adsorbate ratios, the number of available active binding sites is limited, leading to lower adsorption percentages (Ad%). As the adsorbent/adsorbate ratio increased, Ad% increased sharply because of the increased availability of active sites and subsequently reached a plateau, exhibiting only marginal changes. This adsorption behavior was consistent.

Effect of Time

The effect of the interaction between adsorbate and adsorbent was studied from the amount of boron removal by the magnetic nanocomposite as shown in Fig.-7. The data indicated that adsorption gradually increased with time and stabilised after 90 min, suggesting that equilibrium had been established. Consequently, a contact time of 90 min was established as the equilibrium contact time for all subsequent adsorption experiments.¹⁵

Impact of Temperature on Boron Removal Efficiency

The boron removal efficiency was examined in the temperature range of 305.15–338.15 K (Fig.-8). The adsorption capacity increased as the temperature increased. This behaviour suggests that higher temperatures enhance the mobility of boron ions, facilitating their interaction with the adsorbent.

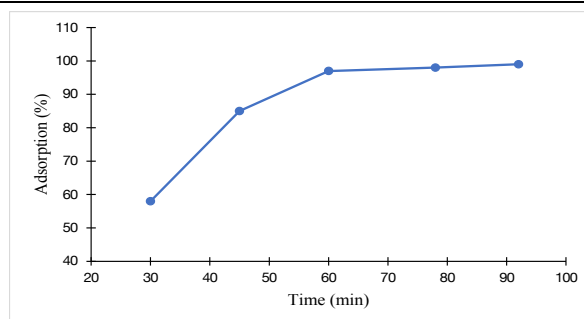


Fig.-7: Removal Efficiency

The reduction in solution viscosity with increasing temperature promoted more efficient transport of boron species to the CM- β -CD-Fe₃O₄ nanoparticle surface. The findings demonstrate that adsorption is positively influenced by temperature, likely owing to the increased kinetic energy and the faster mass transfer of boron ions.

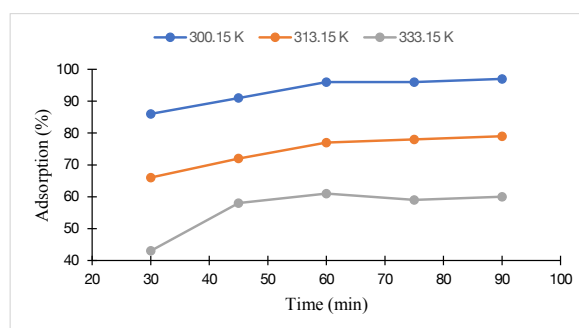


Fig.-8: Effect of Temperature

CONCLUSION

The present study successfully synthesized CM- β -CD-Fe₃O₄ nanoparticles, integrating a magnetic core for rapid phase separation with a functional carboxymethyl- β -cyclodextrin shell. The adsorbent achieved a maximum boron removal efficiency of 98.0% under optimized conditions (pH 5.0, 0.3 g dosage, 50 mg/L boron concentration, and 60 min contact time), consistently outperforming conventional commercial adsorbents. By providing a high-capacity, regenerable solution for wastewater remediation, this research directly contributes to the targets of Sustainable Development Goal 6 (Clean Water and Sanitation). Specifically, the improvement of water quality through the elimination of toxic metalloids. Future research should prioritize the investigation of the efficacy of these nanoparticles in complex industrial effluents and continuous-flow systems to validate their large-scale applicability and long-term stability for practical deployment.

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CONFLICT OF INTERESTS

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

AUTHOR CONTRIBUTIONS

The present work is related to *SDG-6: Clean Water and Sanitation*. All the authors contributed significantly to this manuscript, participated in reviewing/editing and approved the final draft for publication. The research profile of the authors can be verified from their ORCID ids, given below:

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