

Equilibrium, Kinetic, Thermodynamic, and Desorption Studies on the Efficient Uptake of Lead and Copper Ions Using Amino-functionalized Grafted Cross-linked Chitosan Beads.

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Abstract—Heavy metal contamination in water presents significant environmental and health challenges, making effective removal methods essential. This study investigated amino-functionalized grafted cross-linked chitosan beads (Chis_CL_Gr) as an adsorbent for removing Pb^{2+} and Cu^{2+} ions from synthetic wastewater. The chitosan beads were prepared and modified through cross-linking and amino-functionalization, then characterized using Fourier Transform Infrared (FTIR) spectroscopy and Brunauer-Emmett-Teller (BET) analysis to confirm structural and surface properties. Batch adsorption experiments were conducted across varying pH levels, temperatures, and agitation speeds to optimize adsorption conditions. Thermodynamic analysis revealed the adsorption process to be spontaneous and endothermic, while kinetic studies identified a chemisorption mechanism, well-modeled by the pseudo-second-order kinetic equation ($R^2 = 0.99$). Agitation speed improved diffusion and binding efficiency up to a Reynolds number threshold. Langmuir isotherm modeling indicated a monolayer adsorption process, with maximum binding capacities of 376 mg/g for Pb^{2+} and 348 mg/g for Cu^{2+} ions. Regeneration tests demonstrated strong reusability, with high adsorption efficiency up to the fourth cycle before structural degradation affected performance. These findings suggest Chis_CL_Gr beads as a viable, cost-effective solution for heavy metal ion removal in wastewater treatment.

Keywords—Binding capacity, Desorption, Heavy metal ions, Kinetics, Wastewater.

I. INTRODUCTION

In recent years, heavy metal (HM) ion contamination of water bodies has become a significant environmental and health concern [1,2]. Therefore, it is essential to remove these pollutants from the environment [3,4]. Various traditional methods have been employed to mitigate the toxic effects of HM ions, including ion exchange, chemical precipitation, membrane separation, solvent extraction, and adsorption [5]. Among these, adsorption is favored due to its low cost, ease of use, environmental friendliness, and high purification efficiency [6]. However, some methods have limitations; for

instance, solvent extraction requires significant capital investment and complex multistage processes, while chemical precipitation can produce sludge and is less effective for low concentrations of HM ions [7]. Adsorption is widely utilized across industries such as medicine, pharmaceuticals, and environmental management, and ongoing research aims to understand better the interactions at the adsorbent/adsorbate interface [8]. It has proven effective in removing various contaminants from water and wastewater [9,10]. This process involves the attraction and retention of molecules on a solid surface, facilitating the transfer of liquid-phase constituents into the solid phase. Recent advancements have highlighted chitosan as a promising adsorptive material. Derived from the deacetylation of chitin, a naturally occurring polysaccharide found in crustaceans and fungi, chitosan consists of 2-amino-2-deoxy-D-glucopyranose units linked by (1-4) glycosidic bonds [11-13]. Its unique properties, including antimicrobial activity, biocompatibility, non-toxicity, and biodegradability, make it suitable for applications in wastewater treatment, pharmaceuticals, environmental protection, textiles, biotechnology, cosmetics, food processing, and agriculture [9]. Chitosan contains two reactive groups, amino, and hydroxyl, which facilitate graft copolymerization [14]. The amine group at the C₂ position is particularly susceptible to chemical modification due to its lone pair of electrons. Chitosan can be modified physically or chemically to enhance its reactivity, improve adsorption kinetics, or create hybrid materials combining synthetic and natural polysaccharides [15]. Thus, this study aims to develop and optimize chitosan-based adsorbents, specifically cross-linked and grafted chitosan beads, for the effective removal of heavy metal ions from contaminated water while also investigating their adsorption performance, regeneration potential, and structural properties.

II. MATERIALS AND METHODS

A. Adsorbate Preparation Process

The stock mixture used in this study was prepared by separately dissolving $CuSO_4 \cdot 5H_2O$ and $Pb(NO_3)_2$ in distilled water. Subsequently, this stock solution was diluted with

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distilled water to achieve the desired initial concentrations ranging from 40 to 80 mg/L.

B. Chitosan Modification Process

The synthesis of Chis_CL_Gr beads involved a multi-step modification process to enhance the chitosan's adsorption properties. In the first stage, 30 grams of chitosan powder were dissolved in 1 liter of a 5% (v/v) acetic acid solution, which facilitated the complete dissolution of chitosan and increased accessibility to binding sites. In the second stage, a 2.5% glutaraldehyde solution was added to the dissolved chitosan under continuous stirring for two hours with a magnetic stirrer. This step facilitated cross-linking, where glutaraldehyde acted as a cross-linking agent by reacting with the amino groups in chitosan, enhancing the bead stability in acidic conditions.

The cross-linked chitosan solution was converted into bead form in the third stage. This was accomplished by transferring the mixture through a glass pipette, utilizing a peristaltic pump to drop the solution into a 1M sodium hydroxide solution, as illustrated in Fig. 1. The sodium hydroxide induced gelation of the cross-linked chitosan, forming solid Chis_CL beads. Afterward, these beads were thoroughly washed with distilled water to remove residual sodium hydroxide and prepared for the final grafting step, which would complete the formation of Chis_CL_Gr beads with enhanced binding capabilities for heavy metal ions.

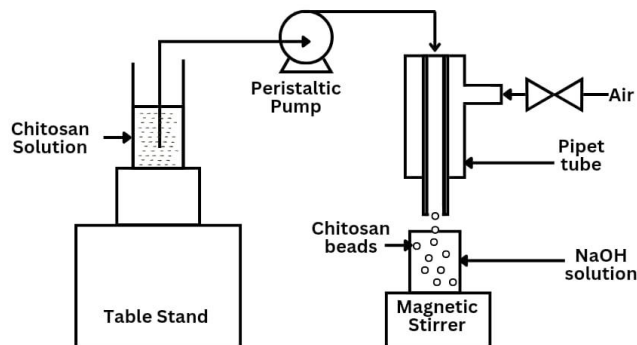


Fig. 1. Process flow diagram illustrating the manufacturing of chitosan beads.

The beads were rinsed with distilled water to remove excess sodium hydroxide. Fourthly, microwave irradiation was used to graft the Chis_CL beads. In a conical flask, 4 g of the Chis_CL beads were combined with 0.5 g/L of 5-amino-1,10-phenanthroline. Then, for 20 minutes, the flask was heated to medium-low power in a microwave. After being cleaned with distilled water, the Chis_CL_Gr beads were prepared for use in the binding experiment. Fig. 2 presents the set of beads made from chitosan powder.

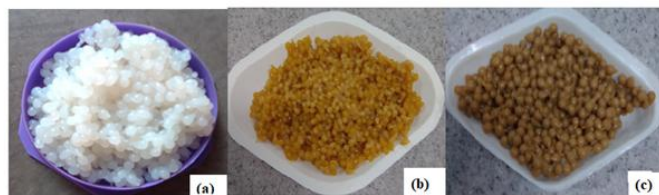


Fig. 2. Sets of produced beads: (a) Chis beads, (b) Chis_CL beads, (c) Chis_CL_Gr beads.

Equation (1) was used to calculate the water content (WC), which aids in transporting adsorbates onto Chis_CL_Gr beads.

$$WC = \frac{W_x - W_y}{W_y} \quad (1)$$

where, W_x and W_y indicate the mass of wet Chis_CL_Gr beads and dry Chis_CL_Gr beads, respectively.

C. Characterization and Analytical Techniques

To thoroughly analyze the structural and surface properties of the three bead variants (Chis, Chis_CL, and Chis_CL_Gr), Fourier Transform Infrared (FTIR) spectroscopy and Brunauer-Emmett-Teller (BET) analysis were employed. FTIR spectra were obtained across a wavelength range of 500–4000 cm^{-1} to identify specific functional groups and assess chemical modifications introduced during cross-linking and grafting. BET analysis, conducted via nitrogen adsorption, provided detailed measurements of surface area, pore volume, and pore size for each bead type, offering insights into their porosity and adsorption potential. Furthermore, adsorption isotherms and kinetic studies were exclusively conducted on Chis_CL_Gr beads to understand their binding behavior with Pb^{2+} and Cu^{2+} ions. These analyses allowed for a comprehensive evaluation of adsorption capacity, rate, and efficiency, ultimately demonstrating the suitability of Chis_CL_Gr beads for metal ion removal in wastewater treatment.

D. Adsorption and Desorption Studies of Pb^{2+} and Cu^{2+} Ions

Batch binding experiments using Chis_CL_Gr beads were conducted to study the adsorption of Pb^{2+} and Cu^{2+} ions under controlled conditions. Metal ion solutions of specific concentrations were adjusted to the desired pH using 0.1 M HCl or NaOH, and the adsorption process was evaluated through kinetic studies at 45°C and 120 rpm. The adsorption capacity (q_e) and percentage removal (%R) were calculated using standard equations. Desorption studies were performed using 0.5 M HCl to regenerate the adsorbent material, and the process was repeated for five cycles to assess reusability. Binding isotherms, including the Langmuir and Freundlich models, were applied to evaluate the adsorption mechanism, with the Langmuir model indicating adsorption on homogeneous surfaces and the Freundlich model accounting for heterogeneous adsorption behavior [16]. Thermodynamic analysis using equilibrium constants allowed for the calculation of entropy and enthalpy, providing insights into the spontaneity of the binding reactions. Adsorption kinetics were analyzed

through pseudo-first and pseudo-second-order models to determine the rate and mechanism of adsorption, while error analysis using Marquardt's percent standard deviation (MPSD) helped validate the accuracy of the models [17].

III. RESULTS AND DISCUSSION

A. FTIR Analysis of Structural and Functional Modifications

The FTIR spectra were utilized for the analysis of the structure and functional groups of Chis, Chis_CL, and Chis_CL_Gr beads, as depicted in Fig. 3. Chis's spectrum exhibited typical features of glycosidic material, including a peak at 3392 cm^{-1} indicating the presence of hydroxyl and amine groups and another peak at 2841 cm^{-1} attributed to $-\text{CH}_2$ stretching. The peaks at 1000 cm^{-1} and 1817 cm^{-1} represented oxygenated functional groups and $\text{C}=\text{O}$ stretching, respectively, while a peak at 779 cm^{-1} was associated with aromatic C-H deformation [18,19]. The cross-linking with glutaraldehyde induced changes in the Chis beads, leading to significant peak shifts, such as from 3392 to 3487 cm^{-1} and 2841 to 2832 cm^{-1} , indicating alterations in chemical bonding. The FTIR spectra of Chis_CL_Gr beads exhibited further peak shifts, including from 3487 to 3332 cm^{-1} and 2840 to 2844 cm^{-1} , as well as the emergence of new peaks at 1377 and 1570 cm^{-1} , corresponding to N-H and C-H bending vibrations. The increased intensity between 1000 and 1500 cm^{-1} implied successful grafting, with OH and NH_2 groups contributing to a high metal-binding capability.

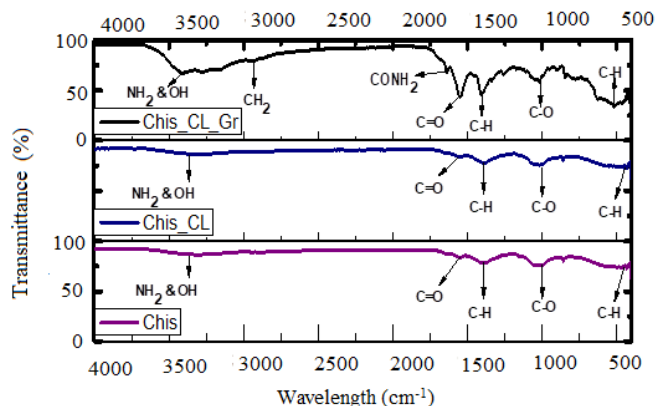


Fig. 3. FTIR of Chis, Chis_CL and Chis_CL_Gr beads, respectively.

IV. BRUNAUER-EMMETT-TELLER SURFACE AREA

The Brunauer-Emmett-Teller (BET) surface area, pore size, and pore volume for Chis, Chis_CL, and Chis_CL_Gr beads, as shown in Table I, indicate that cross-linked beads (Chis_CL) have lower values for these properties. This reduction is likely due to the involvement of chitosan's amine groups in the cross-linking reaction. Conversely, the grafted cross-linked beads (Chis_CL_Gr) demonstrate an increase in BET surface area, pore size, and pore volume, attributed to additional amine groups grafted onto the backbone, which provides more binding sites for lead and copper ions.

TABLE I: BET SURFACE AREA, PORE SIZE AND PORE VOLUME OF CHIS, CHIS_CL AND CHIS_CL_Gr BEADS.

Bead sample	Surface area (m^2/g)	Pore volume (cm^3/g)	Pore size (nm)
Chis	200.26	0.54	50.12
Chis_CL	170.32	0.43	43.59
Chis_CL_Gr	265.78	0.76	69.44

V. EVALUATION OF MODIFIED CHITOSAN BEADS FOR EFFICIENT HEAVY METAL ION ADSORPTION: INFLUENCE OF pH, TEMPERATURE, AND REGENERATION POTENTIAL

The efficiency of modified chitosan beads (Chis_CL_Gr) in binding Pb^{2+} and Cu^{2+} ions from aqueous solutions. The results underscore the critical influence of pH on removal efficiency, with optimal performance achieved at pH 5. At this pH, the conditions favorably align for metal ion binding to the amine groups present on the adsorbent, facilitating effective complexation and attachment of Pb^{2+} and Cu^{2+} ions to the bead surface. The increased availability of free electron pairs on the nitrogen atoms of the amine groups at this pH enhances the adsorption process, creating stronger and more stable interactions between the metal ions and adsorbent. This pH-dependent binding behavior aligns with findings from Fan et al. [20], and is illustrated in Fig. 4., highlighting the significant role that pH conditions play in achieving maximum adsorption efficiency.

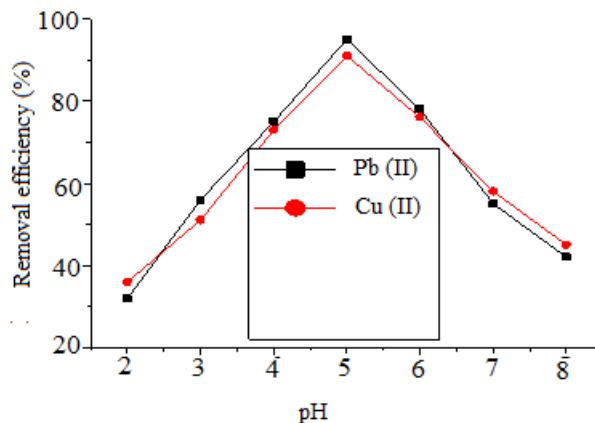


Fig. 4. Effect of pH on percentage removal of Pb^{2+} and Cu^{2+} by Chis_CL_Gr beads (conditions: adsorbent mass: 4 g; contact time: 70 min; Temperature: 45°C ; initial concentration: 40 mg/L, stirring speed: 120 rpm).

The adsorption process was found to be spontaneous and endothermic, with increased removal efficiency at higher temperatures, aligning with the results of Ofomaja and Naidoo [21]. Kinetic analysis revealed that the pseudo-second-order model accurately describes the data ($R^2 = 0.99$), confirming that chemisorption is the primary binding mechanism [22]. Additionally, the agitation speed significantly impacted both external and intra-particle diffusion processes. Increased agitation speed led to greater turbulence in the solution, which enhanced the distribution of solute particles around the

adsorbent surface and reduced the thickness of the boundary layer. This improvement in turbulence effectively promoted the binding of Pb^{2+} and Cu^{2+} ions to the adsorbent, as shown in Fig. 5. However, the beneficial effects of increased agitation were observed only up to a specific Reynolds number threshold, beyond which no further enhancement in adsorption efficiency was detected. This threshold suggests that, while external film mass transfer initially limits the adsorption rate, the process reaches a point where external turbulence no longer contributes to additional binding efficiency [23].

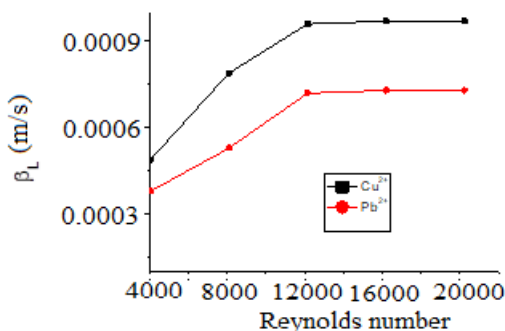


Fig. 5. Relationship between external diffusion coefficient and Reynolds number for adsorbates adsorption onto Chis_CL_Gr beads.

Langmuir isotherm modeling confirmed that adsorption onto the Chis_CL_Gr beads follows a monolayer adsorption mechanism. This model implies that metal ions occupy individual, uniform binding sites on the adsorbent surface without interaction between adsorbed molecules, maximizing surface coverage. The results demonstrated optimal binding strength at temperatures up to $45^{\circ}C$, where the adsorbent exhibits its highest binding capacity. However, as temperatures exceed $45^{\circ}C$, a gradual decline in adsorption capacity is observed, likely due to thermal disruption of adsorbent-metal ion interactions and potential changes in the adsorbent structure, as previously noted by Xu et al. (2022).

The adsorbent's reusability was evaluated through regeneration studies, as shown in Fig. 6. These studies reveal that the Chis_CL_Gr beads maintained high efficiency and binding capacity across repeated adsorption-desorption cycles, with minimal capacity loss up to the fourth cycle. This stable performance highlights the structural robustness of the modified chitosan beads, making them suitable for repeated use. Beyond the fourth cycle, a noticeable reduction in binding capacity occurs, suggesting some structural degradation, likely from repeated exposure to desorbing agents. This could weaken the functional groups responsible for metal binding. These findings underscore the Chis_CL_Gr beads' reusability potential for effective and sustainable heavy metal ion removal, particularly within moderate regeneration cycles.

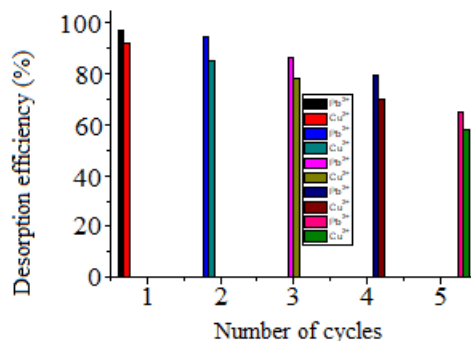


Fig. 6. The plot of multiple cycles of adsorption and desorption.

During the initial cycles, the Chis_CL_Gr beads effectively adsorbed Pb^{2+} and Cu^{2+} ions, maintaining stable performance with negligible mass loss. However, a noticeable decline in adsorption efficiency was observed beyond the fourth regeneration cycle. This reduction is primarily attributed to structural degradation of the chitosan beads, likely due to repeated acid treatment and mechanical agitation, which compromise the integrity of the amine groups essential for metal ion binding [24]. Consequently, the beads exhibit lower adsorption capacity and a substantial mass loss by the fifth cycle, indicating that the material's stability and durability are critical factors in its suitability for extended use. These findings underscore the need for further optimization of the regeneration process or modification of the adsorbent structure to enhance its robustness for sustainable, long-term application in wastewater treatment.

VI. CONCLUSION

Amino-functionalized grafted cross-linked chitosan beads (Chis_CL_Gr) proved highly effective for the adsorption of Pb^{2+} and Cu^{2+} ions from synthetic wastewater, demonstrating their potential as a cost-effective and sustainable solution for heavy metal removal. The study found optimal adsorption occurred at pH 5, leveraging strong interactions between the metal ions and amine groups on the adsorbent. Thermodynamic analysis confirmed the process as spontaneous and endothermic, with efficiency increasing at elevated temperatures, while kinetic modeling indicated a chemisorption mechanism best fit by the pseudo-second-order model. Furthermore, the Langmuir isotherm supported monolayer adsorption with high maximum binding capacities, while regeneration tests showed strong reusability up to the fourth cycle, after which structural degradation impacted performance. These findings underscore the practical applicability of Chis_CL_Gr beads for effective wastewater treatment, although further optimization may be needed to enhance long-term stability across multiple regeneration cycles.

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