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RESEARCH PAPER

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Extraction, characterization and adsorptive removal of copper and zinc from industrial effluents using shrimp shell-derived chitosan

P. Raja Rajeswari, R. Rajakumar*

Post graduate and Research Department of Biotechnology, Maruthupandiyar College, Thanjavur, Affiliated to Bharathidasan University, Tiruchirappalli, Tamil Nadu, India

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ABSTRACT

The present study investigated the potential of shrimp shell-derived chitosan as an eco-friendly bio sorbent for the removal of copper (Cu^{2+}) and zinc (Zn^{2+}) from industrial effluents. Shrimp shell waste collected from seafood processing industries in the Thoothukudi district, Tamil Nadu, India, was processed to extract chitin and subsequently converted into chitosan through sequential demineralization, deproteinization and microwave-assisted deacetylation. The extracted chitosan was characterized by determining its moisture content, ash content, pH, viscosity, degree of deacetylation and FTIR spectral characteristics. Batch adsorption experiments were performed to evaluate the influence of pH, adsorbent dosage, and temperature on the removal efficiency of copper and zinc. The extracted chitosan exhibited favorable physicochemical properties, including a moisture content of 8.5%, ash content of 0.78%, viscosity of 310 cP, pH of 6.4, and a degree of deacetylation of 87.32%. FTIR analysis confirmed the presence of characteristic amino and hydroxyl functional groups responsible for metal adsorption. Industrial effluents contained measurable concentrations of copper (0.42 ± 0.03 mg L^{-1}), zinc (0.68 ± 0.04 mg L^{-1}), and lead (0.12 ± 0.01 mg L^{-1}), while cadmium was below the detection limit. Maximum adsorption efficiencies of 89.97% for copper and 96.36% for zinc were achieved under optimized conditions. Adsorption efficiency increased with adsorbent dosage but decreased with increasing temperature, indicating that the adsorption process was favorable under moderately acidic to neutral pH and lower temperature conditions. The findings demonstrate that shrimp shell-derived chitosan is an efficient, biodegradable, and low-cost bio sorbent for the removal of copper and zinc from industrial wastewater.

***Corresponding Author:** R. Rajakumar ✉ biotechrajakumar@gmail.com

INTRODUCTION

Heavy metal contamination has become one of the most significant environmental challenges affecting aquatic ecosystems worldwide. Unlike many organic pollutants, heavy metals are non-biodegradable, persistent, and capable of accumulating in aquatic environments for prolonged periods. Their continuous release into coastal waters results in bioaccumulation within aquatic organisms and biomagnification through the food chain, ultimately posing serious ecological and human health risks. Among the various heavy metals, copper (Cu) and zinc (Zn) are essential micronutrients at trace concentrations but become toxic when present in excessive amounts, causing oxidative stress, metabolic dysfunction, impaired growth, and reduced reproductive capacity in aquatic organisms (Islam *et al.,* 2023; Srinivasan *et al.,* 2023).

Coastal ecosystems are particularly vulnerable to heavy metal pollution because they receive contaminants from multiple terrestrial and marine sources. Although natural processes such as rock weathering, volcanic emissions, hydrothermal activities, and riverine transport contribute to the background concentrations of metals in marine environments, these inputs are generally regulated by natural geochemical cycles. In contrast, rapid industrialization, urban expansion, and intensive coastal development have substantially increased anthropogenic metal loading, making human activities the predominant source of heavy metal contamination in most coastal regions (Yadav *et al.,* 2019). Industrial effluents discharged from electroplating, chemical manufacturing, fertilizer production, textile processing, thermal power plants, mining, and metallurgical industries represent major contributors of copper and zinc pollution. Additional inputs arise from agricultural runoff, municipal wastewater, shipping activities, atmospheric deposition, and urban stormwater, leading to elevated metal concentrations in coastal waters, sediments, and aquatic organisms (El-Sharkawy *et al.,* 2025).

The persistence of heavy metals in the environment limits the effectiveness of natural attenuation processes and presents considerable challenges for conventional wastewater treatment technologies. Physical and chemical remediation methods, including chemical precipitation, ion exchange, membrane filtration, reverse osmosis, and electrochemical treatments, are often associated with high operational costs, energy-intensive processes, incomplete metal removal at low concentrations, and the generation of secondary pollutants such as toxic sludge. These limitations have stimulated growing interest in sustainable and environmentally friendly alternatives that are both efficient and economically feasible (Syed *et al.,* 2022).

Among emerging remediation approaches, biosorption has received considerable attention as a green technology for the removal of toxic metals from contaminated water. Biosorption utilizes naturally occurring biological materials possessing functional groups capable of binding dissolved metal ions through mechanisms such as ion exchange, complexation, chelation, electrostatic attraction, and surface adsorption. Compared with conventional treatment methods, biosorption offers several advantages, including high metal removal efficiency, low operating costs, minimal chemical requirements, reduced sludge production, and compatibility with environmentally sustainable treatment systems (Crini *et al.,* 2019).

Chitosan, a deacetylated derivative of chitin, has emerged as one of the most effective natural biosorbents for heavy metal remediation. Chitin is the second most abundant renewable biopolymer after cellulose and occurs naturally in the exoskeletons of crustaceans, insect cuticles, and fungal cell walls. Through controlled deacetylation, chitin is converted into chitosan, a biopolymer characterized by abundant amino and hydroxyl functional groups that exhibit strong affinity toward divalent and multivalent metal ions. These reactive functional groups enable chitosan to efficiently adsorb heavy metals such as copper, zinc, cadmium, lead, chromium, and nickel under a wide range of environmental conditions. In addition to its excellent adsorption capacity, chitosan is biodegradable, biocompatible, non-toxic, renewable, and readily modifiable, making it an attractive material for sustainable wastewater treatment applications (Reddy *et al.,* 2023; Kumar *et al.,* 2022).

The utilization of shrimp shell waste as a raw material for chitosan production further enhances the environmental significance of this approach. Seafood processing industries generate substantial quantities of crustacean shell residues each year, and their improper disposal contributes to odor problems, microbial proliferation, and environmental pollution. Transforming shrimp shell waste into value-added chitosan not only addresses waste management concerns but also supports circular economy principles by converting an abundant biological by-product into an efficient biosorbent for environmental remediation. This strategy simultaneously promotes resource recovery, waste valorization, and sustainable pollution control while reducing dependence on expensive synthetic adsorbents (Ghimire *et al.,* 2022; Meenalosini *et al.,* 2020).

Despite numerous reports describing the adsorption properties of commercial chitosan, relatively few studies have investigated the production of chitosan from locally available shrimp shell waste and its application for the simultaneous removal of copper and zinc from industrial effluents. Furthermore, systematic optimization of adsorption parameters and comprehensive characterization of the extracted biopolymer remain essential for improving its practical applicability in wastewater treatment.

Therefore, the present study aims to extract chitosan from shrimp shell waste generated by seafood processing industries, characterize its physicochemical properties, and evaluate its adsorption efficiency for the removal of copper and zinc from industrial effluents under optimized operating conditions. By integrating seafood waste valorization with sustainable heavy metal remediation, this study contributes to the development of environmentally friendly and economically viable technologies for controlling industrial metal pollution while supporting circular bioeconomy initiatives.

MATERIALS AND METHODS

Collection and preparation of shrimp shell waste

Shrimp shell waste was collected from seafood processing industries located in the Thoothukudi district, Tamil Nadu, India. The collected material consisted mainly of shrimp exoskeletons, cephalothorax, and tail portions generated during seafood processing. The samples were transported to the laboratory in sterile polyethylene bags and processed immediately to prevent microbial degradation. The shells were thoroughly washed with running tap water to remove adhering tissues and impurities, followed by repeated rinsing with distilled water to eliminate residual salts. The cleaned shells were shade-dried for 48 h and subsequently sun-dried until constant weight was attained. The dried shells were pulverized using a laboratory grinder and sieved to obtain a uniform particle size. The powdered material was stored in airtight containers at room temperature until further use.

Extraction of chitin and preparation of chitosan

Demineralization

Chitin was extracted from shrimp shell powder using sequential demineralization and deproteinization, followed by deacetylation to obtain chitosan. For demineralization, the powdered shells were treated with 5% (v/v) hydrochloric acid at a solid-to-liquid ratio of 1:6 (w/v) and incubated at room temperature for 24 h with intermittent stirring. The demineralized residue was washed repeatedly with distilled water until neutral pH was attained and then dried at 60°C (Kumari *et al.,* 2015).

Deproteinization

The demineralized material was subsequently subjected to deproteinization using 5% (w/v) sodium hydroxide at a solid-to-liquid ratio of 1:10 (w/v). The suspension was maintained at 60–70°C for 48 h with continuous stirring to facilitate protein removal. Following alkali treatment, the residue was thoroughly washed with distilled water to neutral pH and dried at 60°C to obtain purified chitin (Arbia *et al.,* 2013).

Deacetylating

Chitosan was prepared by deacetylating the purified chitin using 60% (w/v) sodium hydroxide under microwave-assisted heating for 2 h. The reaction mixture was cooled to room temperature, washed repeatedly with distilled water until neutral pH was achieved, dried at 60°C, ground into a fine powder, and stored in airtight containers for subsequent characterization and adsorption studies (Khan *et al.,* 2022).

Physicochemical characterization of chitosan

The extracted chitosan was characterized to determine its physicochemical properties. Moisture content was estimated gravimetrically by drying the samples at 105°C until constant weight was achieved (Huang *et al.,* 2023). Ash content was determined by incinerating approximately 1 g of chitosan in a muffle furnace at 600°C for 4 h (Kumari *et al.,* 2015). The pH was measured using a calibrated digital pH meter after dissolving 1% (w/v) chitosan in 1% (v/v) acetic acid. Viscosity was determined using an Ostwald viscometer at room temperature with a 1% chitosan solution (Kayalvizhi and Evany nithya, 2019). Functional groups present in the extracted chitosan were analyzed using Fourier Transform Infrared (FTIR) spectroscopy over the spectral range of 4000–400 cm^{-1} (Huang *et al.,* 2023). The degree of deacetylation was determined by the acid-base titration method using standardized hydrochloric acid and sodium hydroxide solutions (Kyzas and Bikiaris, 2015).

Collection of industrial effluents

Industrial wastewater samples were collected from selected effluent discharge points in the Thoothukudi industrial region. Sampling locations included industries associated with metal processing, chemical manufacturing, thermal power generation, and other industrial activities. Samples were collected in pre-cleaned acid-washed polyethylene containers, transported to the laboratory under refrigerated conditions (4°C), and analyzed within 24 h.

Characterization of industrial effluents

Physicochemical parameters including pH, electrical conductivity, total dissolved solids, colour, and odour were determined following standard analytical procedures. Heavy metals were quantified after acid digestion using concentrated nitric acid and perchloric acid. The digested samples were filtered and analyzed for copper and zinc concentrations using Atomic Absorption Spectrophotometry (AAS) with certified reference standards (APHA, 2017). All measurements were performed in triplicate, and reagent blanks were included for quality assurance.

Batch adsorption experiments

Batch adsorption experiments were conducted to evaluate the efficiency of the extracted chitosan for copper and zinc removal. Synthetic aqueous solutions containing known concentrations of Cu^{2+} and Zn^{2+} ions (10–120 mg L^{-1}) were prepared using analytical-grade metal salts dissolved in deionized water. A predetermined amount of chitosan (50–250 mg) was added to 100 mL of metal solution in 250 mL Erlenmeyer flasks and agitated at 100 rpm using an orbital shaker (Crini *et al.,* 2019).

The influence of operational parameters including solution pH (2–8), temperature (25–45°C), adsorbent dosage, contact time, and initial metal concentration on adsorption efficiency was investigated by varying one parameter while maintaining the remaining variables constant. Following equilibration, the suspensions were filtered through Whatman No. 1 filter paper, and the residual concentrations of copper and zinc were determined by Atomic Absorption Spectrophotometry. The percentage removal efficiency and adsorption capacity were calculated using standard adsorption equations. All experiments were conducted in triplicate, and the results are expressed as mean ± standard deviation (Khan *et al.,* 2022).

Statistical analysis

All experimental analyses were performed in triplicate, and the data are presented as mean ± standard deviation. Statistical analysis was carried out using SPSS software (IBM SPSS Statistics, Version 26.0, IBM Corp., Armonk, NY, USA). Significant differences among treatments were evaluated using one-way analysis of variance (ANOVA), followed by Tukey's multiple comparison test at a significance level of $p < 0.05$.

RESULTS

Extraction and characterization of chitosan

Shrimp shell waste collected from seafood processing industries in the Thoothukudi district served as an abundant source of chitin for chitosan production. Sequential demineralization, deproteinization, and deacetylation successfully yielded high-quality chitosan with desirable physicochemical characteristics. During demineralization, vigorous effervescence indicated effective removal of calcium carbonate, whereas alkaline treatment efficiently eliminated proteinaceous materials, producing a clean fibrous chitin matrix. Microwave-assisted deacetylation resulted in a cream-coloured chitosan powder that dissolved readily in dilute acetic acid, indicating successful conversion of chitin into chitosan. The extracted chitosan exhibited favorable physicochemical properties for adsorption applications. Moisture and ash contents were 8.5% and 0.78%, respectively, indicating low residual mineral content and good storage stability. The pH of the chitosan solution was 6.4, while the viscosity was 310 cP, suggesting moderate molecular weight and adequate polymer integrity. The degree of deacetylation was determined to be 87.32 ± 1.1%, confirming efficient removal of acetyl groups and the generation of abundant free amino groups responsible for metal binding (Table 1).

Table 1. Physicochemical properties of extracted chitosan

Sl	Physicochemical properties	Experimental result
1.	Chitosan yield (%)	25.6 ±0.56
2.	Moisture Content (%)	8.5 ±0.2
3.	Ash Content (%)	0.78 ±0.05
4.	pH of Solution	6.4 ±0.05
5.	Viscosity (cP or relative flow time)	310.5 ±9.3
6.	Degree of Deacetylation (DD, %)	87.32 ±1.12
7.	Solubility in 1% acetic acid (%)	97.8 ±0.52
8.	Molecular weight (kDa)	243.1 ±12.2
9.	Colour	Cream white
10.	Appearance	Fine powder

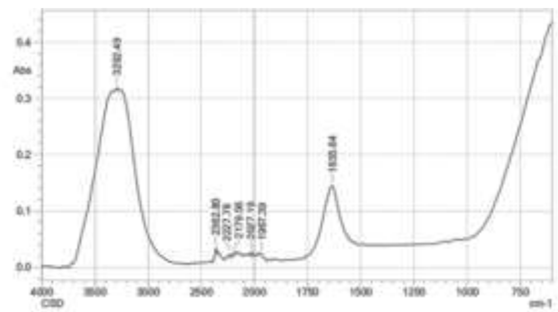

Fig. 1. FTIR spectrum of chitosan extracted from shrimp shell waste

FTIR analysis

FTIR analysis further confirmed the successful preparation of chitosan. A broad absorption band at approximately 3450 cm^{-1} corresponded to overlapping O–H and N–H stretching vibrations, whereas the band at 2926 cm^{-1} represented C–H stretching of the polysaccharide backbone. Weak amide I (1650 cm^{-1}) and amide II (1556 cm^{-1}) bands demonstrated extensive deacetylation. Strong absorption peaks within the 1000–1200 cm^{-1} region and the characteristic β-(1→4) glycosidic linkage at approximately 895 cm^{-1} confirmed preservation of the chitosan polymer structure (Fig. 1).

Physicochemical characteristics of industrial effluents

Industrial wastewater collected from selected discharge points in the Thoothukudi industrial region exhibited characteristics typical of industrial contamination. The wastewater was light brown with a slightly unpleasant odour and showed a slightly acidic pH (6.4 ± 0.1). Total dissolved solids and electrical conductivity were recorded as 1120 ± 45 mg L^{-1} and 1.82 ± 0.06 mS cm^{-1}, respectively, indicating a considerable dissolved ionic load (Table 2).

Table 2. Physico chemical analysis of industrial effluent

Sl	Parameter	Method used	Unit	Observed value (Mean ± SD)	Permissible limit
1	pH	Digital pH meter	—	6.4 ± 0.1	6.5–8.5
2	Colour	Visual comparison	—	Light brown	Colourless
3	Odour	Sensory analysis	—	Slightly unpleasant	Odourless
4	Total Dissolved Solids (TDS)	Gravimetric method	mg L⁻¹	1120 ± 45	2000
5	Electrical Conductivity	Conductivity meter	mS cm⁻¹	1.82 ± 0.06	—
6	Copper (Cu^{2+})	AAS	mg L⁻¹	0.42 ± 0.03	1.0
7	Zinc (Zn^{2+})	AAS	mg L⁻¹	0.68 ± 0.04	5.0
8	Lead (Pb^{2+})	AAS	mg L⁻¹	0.12 ± 0.01	0.10
9	Cadmium (Cd^{2+})	AAS	mg L⁻¹	BDL	0.01

Heavy metal analysis

Heavy metal analysis revealed detectable concentrations of copper, zinc, and lead in all samples. Copper and zinc concentrations were 0.42 ± 0.03 mg L^{-1} and 0.68 ± 0.04 mg L^{-1}, respectively, whereas lead concentration reached 0.12 ± 0.01 mg L^{-1}. Cadmium remained below the detection limit throughout the study. Although copper and zinc concentrations were within permissible discharge limits, the presence of these metals indicates continuous industrial inputs into the receiving aquatic environment (Table 2).

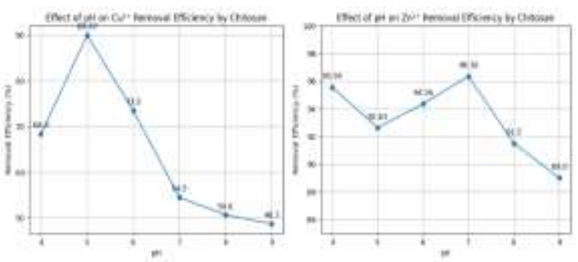

Fig. 2. Effect of pH on the removal efficiency of Cu^{2+} and Zn^{2+} by chitosan

Adsorption of copper and zinc by chitosan

Effect of pH

Solution pH markedly influenced the adsorption of both copper and zincs. Copper adsorption increased from 68.30% at pH 4 to a maximum removal efficiency of 89.97% at pH 5, after which adsorption progressively declined with increasing pH. In contrast, zinc adsorption remained consistently high across the tested pH range and reached a maximum removal efficiency of 96.36% at pH 7. These results demonstrate that the optimum pH differed between the two metals, with acidic conditions favoring copper adsorption and near-neutral conditions favoring zinc adsorption (Fig. 2).

Effect of adsorbent dosage

Increasing chitosan dosage significantly improved metal removal efficiency. Copper removal increased from 70.40% at 50 mg adsorbent to 89.30% at 200 mg, whereas zinc removal increased from 90.40% to 95.40% over the same dosage range. No appreciable improvement was observed beyond 200 mg, indicating saturation of available adsorption sites under the experimental conditions (Fig. 3).

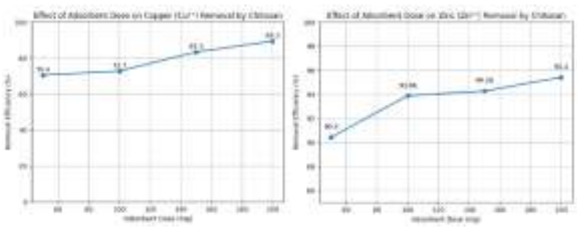

Fig. 3. Effect of adsorbent dose on Cu^{2+} and Zn^{2+} removal by chitosan

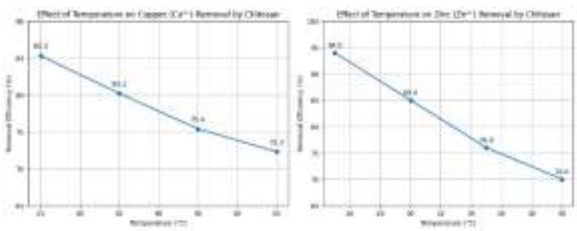

Fig. 4. Effect of temperature on the removal efficiency of Cu^{2+} and Zn^{2+} by chitosan

Effect of temperature

Temperature exerted a negative influence on metal adsorption. Copper removal efficiency decreased from 85.30% at 25°C to 72.30% at 55°C, while zinc removal declined from 94.00% at 25°C to 70.00% at 40°C. The reduction in adsorption efficiency with increasing temperature suggests that the adsorption process is favoured under lower temperature conditions (Fig. 4).

Table 3. Optimum operational conditions for Cu^{2+} and Zn^{2+} removal by shrimp shell-derived chitosan

Metal Ion	Optimum pH	Temperature (°C)	Adsorbent dosage (mg)	Maximum removal efficiency (%)
Cu^{2+}	5	25	200	89.97 ± 0.58
Zn^{2+}	7	25	200	96.36 ± 0.42

Values are expressed as Mean ± Standard Deviation (n = 3).

Optimum adsorption performance

Among the operational variables investigated, solution pH and adsorbent dosage exerted the greatest influence on metal removal efficiency. Maximum copper removal (89.97%) was achieved at pH 5 using 200 mg of chitosan, whereas maximum zinc removal (96.36%) was obtained at pH 7 with the same adsorbent dosage. The high adsorption efficiencies obtained for both metals demonstrate the effectiveness of shrimp shell-derived chitosan as a low-cost biosorbent for the removal of copper and zinc from contaminated aqueous systems (Table 3).

DISCUSSION

The present study demonstrated that shrimp shell waste generated from seafood processing industries can be effectively converted into high-quality chitosan suitable for heavy metal remediation. The sequential demineralization, deproteinization, and microwave-assisted deacetylation procedures yielded chitosan possessing favourable physicochemical characteristics and high adsorption efficiency toward copper and zinc ions. The results highlight the potential of converting seafood processing waste into an environmentally sustainable biosorbent for wastewater treatment, thereby supporting circular bioeconomy and waste valorization strategies.

The low ash content (0.78%) obtained in the present study indicates efficient removal of inorganic minerals during the demineralization process. Similarly, the moisture content (8.5%) was within the acceptable range reported for commercial adsorption-grade chitosan, suggesting good storage stability and minimal hygroscopicity. The degree of deacetylation (87.32%) further confirmed successful conversion of chitin into chitosan. A higher degree of deacetylation increases the number of free amino groups available for metal complexation and is considered one of the most important factors governing the adsorption performance of chitosan. Similar observations have been reported by recent investigations, which demonstrated that chitosan with a degree of deacetylation exceeding 80% exhibits significantly improved adsorption capacity toward divalent metal ions due to enhanced availability of protonatable amino groups and increased surface reactivity (Kumar *et al.,* 2023; Wang *et al.,* 2024).

The FTIR spectrum confirmed the successful preparation of chitosan through the presence of characteristic hydroxyl, amino, and glycosidic functional groups. The reduced intensity of amide I and amide II bands indicated extensive removal of acetyl groups, whereas the broad absorption band corresponding to hydroxyl and amino groups demonstrated the presence of active functional sites responsible for metal binding. These functional groups interact with metal ions through coordination, chelation, ion exchange, and electrostatic attraction. Similar FTIR characteristics have been consistently reported for chitosan-based biosorbents developed from crustacean shell waste for environmental remediation (Zhang *et al.,* 2024; Li *et al.,* 2024).

Industrial wastewater collected from the Thoothukudi industrial region exhibited measurable concentrations of copper, zinc, and lead, indicating continuous anthropogenic metal inputs into the aquatic environment. Although copper and zinc concentrations remained below the prescribed discharge limits, their persistent occurrence suggests long-term accumulation in receiving ecosystems. Lead concentrations marginally exceeded the permissible limit, emphasizing the need for continuous monitoring and effective treatment prior to discharge.

Similar findings have been reported from industrialized coastal regions, where persistent heavy metal contamination contributes to sediment pollution, bioaccumulation and biomagnification in aquatic organisms, habitat degradation, biodiversity loss, and impaired ecosystem functioning (El-Sharkawy *et al.*, 2025).

Solution pH significantly influenced the adsorption of both copper and zinc, although the optimum pH differed between the two metals. Copper adsorption reached its maximum at pH 5, whereas zinc adsorption was highest at pH 7. The observed pH dependency can be explained by changes in the protonation state of amino groups present on the chitosan surface. Under highly acidic conditions, competition between hydrogen ions and metal ions reduces the availability of adsorption sites, whereas increasing pH enhances deprotonation of amino groups, thereby improving metal complexation. At alkaline pH values, the decline in adsorption may be attributed to hydrolysis or precipitation of metal hydroxides, which limits the availability of free metal ions for adsorption. Similar pH-dependent adsorption behaviour has been reported for chitosan and other amino-functionalized biosorbents used for copper and zinc removal (Ahmed *et al.,* 2022; Zhang *et al.,* 2024).

Adsorbent dosage also exerted a significant influence on adsorption efficiency. Increasing the amount of chitosan progressively enhanced the removal of both copper and zinc until equilibrium was reached at an adsorbent dosage of 200 mg. This improvement is primarily associated with an increase in the total surface area and the number of available active adsorption sites. Beyond the optimum dosage, the increase in removal efficiency became negligible, suggesting saturation of binding sites and possible aggregation of adsorbent particles, which reduces the effective surface area. Similar trends have been widely reported in adsorption studies involving shrimp shell-derived chitosan and other biopolymer-based adsorbents (Gao *et al.,* 2023).

Temperature exhibited an inverse relationship with adsorption efficiency, with maximum removal observed at 25°C for both copper and zinc. The reduction in adsorption capacity at higher temperatures indicates that the adsorption process is predominantly exothermic. Elevated temperatures may weaken interactions between metal ions and functional groups or increase the tendency of adsorbed ions to desorb from the chitosan surface. Comparable temperature-dependent adsorption behaviour has been observed in previous investigations evaluating the removal of heavy metals using natural polysaccharide-based biosorbents (Chen *et al.,* 2023).

Overall, zinc removal efficiency was consistently higher than copper removal under the optimized experimental conditions. This difference may be associated with variations in ionic radius, hydration energy, coordination behaviour, and affinity of the respective metal ions toward amino and hydroxyl groups of chitosan. The high removal efficiencies obtained in the present investigation (89.97% for copper and 96.36% for zinc) compare favourably with those reported for commercially available chitosan and several modified biosorbents. These findings indicate that shrimp shell-derived chitosan possesses excellent adsorption performance despite being produced from inexpensive seafood processing waste.

CONCLUSION

The present study concluded that locally produced chitosan derived from shrimp shell waste represents a sustainable, biodegradable, and economically viable biosorbent for industrial wastewater treatment. In addition to reducing heavy metal concentrations in contaminated water, this approach contributes to the valorization of seafood processing residues, thereby minimizing environmental burdens associated with shell waste disposal. The integration of waste recycling with wastewater remediation supports the principles of green chemistry and circular bioeconomy, making shrimp shell-derived chitosan a promising material for large-scale environmental applications.

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