



Preparation, Characterisation, and Performance Evaluation of Organic-Based Adsorbents Derived from Shrimp Shells for the Removal of Heavy Metals from Simulated Power Plant Wastewater: An Applied Study in Basrah

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Article Info	ABSTRACT
Article type: Research Article	This research aims to develop two low-cost, eco-friendly bio-adsorbent materials de-rived from chitosan extracted from shrimp shells, for the removal of carcinogenic heavy ele-ments (lead, Pb^{2+}; nickel, Ni^{2+}; and vanadium, V^{5+}) from wastewater generated by power plants. Adsorption experiments were conducted using multi-metal synthetic aqueous solutions contain-ing Pb^{2+}, Ni^{2+}, and V^{5+} simultaneously, prepared in the laboratory to simulate the reported composition of Basrah power-plant wastewater; real power-plant water was not tested in this study. Chitosan was obtained from shrimp shells via demineralization, deproteinization, and deacety-lation, then crosslinked into two hydrogels: one using natural tannic acid and one using bio-gelatin, to enhance their mechanical and chemical properties. Both materials were character-ized using FTIR, XRD, SEM, BET, and TGA techniques to assess chemical and crystalline structure, porosity, and thermal stability. Both hydrogels exhibited high adsorption efficiency under optimized conditions of pH = 5, contact time of 90 minutes, initial pollutant concentra-tion of 100 mg/L, and temperature of 25°C. For the tannic acid-crosslinked chitosan hydrogel, the maximum removal efficiencies were as follows: lead 98.3%, nickel 91.4%, and vanadium 88.7%. The gelatin-crosslinked hydrogel achieved removal efficiencies of lead 88.6%, nickel 83.2%, and vanadium 79.4%, indicating a slight superiority of the tannic acid-crosslinked hydrogel. Removal efficiency declined above an initial concentration of 150 mg/L but remained stable between pH 5 and 6. These findings demonstrate the potential of both materials, particularly the tannic acid-crosslinked hydrogel, as sustainable, low-cost filters for treating heavy-metalcontaminated water in Basrah Governorate, where deteriorating water quality is linked to elevated pollution-related cancer incidence.
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INTRODUCTION

Wastewater from thermal power plants constitutes a major source of environmental pollution by heavy elements, particularly lead, nickel, and vanadium, which are toxic and carcinogenic according to the International Agency for Research on Cancer (IARC) classification (Obodovych et al., 2023; Volkov et al., 2022; International Agency for Research on Cancer, 2020). These elements cause serious health effects such as neuropathy, kidney disorders, and cardiovascular diseases (Roy et al., 2024). In Iraq, specifically in Basrah Governorate, water pollution levels have intensified due to industrial waste and power plants, leading to an unprecedented rise in

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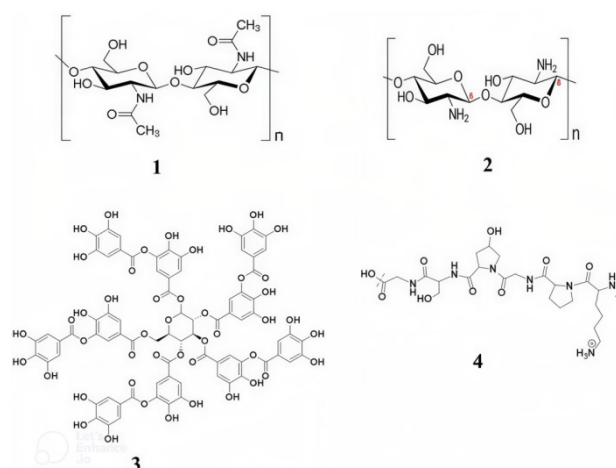


Fig. 1. Chemical structure of (1) Chitin, (2) Chitosan, (3) Tannic acid, and (4) Gelatin

cancer cases and chronic diseases (Salah et al., 2025; Todd, 2024; Abdul & Hameed, 2024).

Interest has recently grown in using low-cost biological materials like chitosan as effective adsorbents for heavy metal removal. Chitosan is a natural polymer derived from chitin (Figure 1) found in crustacean shells, distinguished by its multiple functional groups ($-\text{NH}_2$ and $-\text{OH}$) that contribute to ionic binding with heavy metals (Tsauria et al., 2025; Dambuza et al., 2025). However, the efficiency of raw chitosan may be limited due to its high solubility, prompting researchers to develop chitosan hydrogels using natural crosslinkers such as tannic acid or gelatin to enhance mechanical properties and thermal stability (Sesia et al., 2022; Farasati Far et al., 2024).

FAO and UNEP reports confirm that Basrah produces thousands of tons of marine waste annually, including shrimp shells, which are wasted without utilization, opening opportunities for sustainable environmental applications based on the circular economy (FAO & UNEP, 2022). Additionally, reliance on local resources contributes to cost reduction and achieves environmental and economic sustainability in polluted water treatment projects (Tomičić et al., 2026).

Based on this background, the objective of this study is to prepare and characterize two chitosan hydrogel materials derived from shrimp shell-extracted chitosan, using both tannic acid and gelatin as natural crosslinkers, and to evaluate their efficiency in adsorbing heavy elements (Pb, Ni, V) under realistic operational conditions representing the environment of Basrah Governorate. This study is expected to provide an effective applied model for reusing marine waste and achieving sustainable water management. Recent years have witnessed a significant increase in research concerned with using chitosan and its derivatives as effective adsorbents for removing heavy elements from industrial water, owing to their distinctive chemical and physical properties (Tsauria et al., 2025). Marzuki et al. (2019) used shrimp chitosan to remove Pb and Ni with efficiencies of 59% and 45%, respectively. Al-Hazmi et al. (2025) conducted a study to develop hydrogel beads of Ce-MOF embedded in a crosslinked chitosan-CMC algal matrix, achieving a maximum Ni(II) adsorption capacity of 301.05 mg/g at pH 5 and a dosage of 0.02 g/25 mL. The process followed the Langmuir model and second-order kinetics, was endothermic and spontaneous, and demonstrated stable performance over 6 reuse cycles. While Omidinasab et al. (2018) demonstrated that Fe_3O_4 -CSN effectively removed 99.9% of V(V) and 92.3% of Pd(II) through a highly efficient physicochemical and exothermic adsorption process, Lone et al. (2019) and Badawi et al. (2017) developed chitosan hydrogels crosslinked with tannic acid or gelatin and showed highly promising results under optimized conditions. Raji et al. (2023) highlighted the influence of pH and contact time on adsorption performance and

emphasized the importance of studying initial concentration in reducing active site saturation. At the local level, Salah et al. (2025) and Jassim et al. (2024) confirmed that Basrah's wastewater contains high concentrations of heavy metals, advocating for the development of local solutions based on environmental resources. Despite the abundance of studies addressing chitosan, few have clearly compared two types of hydrogels under standardized operational conditions and within an Iraqi environment. However, many of these previously reported chitosan-based adsorbents display practical limitations that restrict their real-world deployment, including low mechanical stability under continuous-flow operation, limited regeneration and reusability over repeated adsorption–desorption cycles, comparatively modest adsorption capacities relative to engineered sorbents, and reduced performance when applied to real, multi-ion wastewater matrices rather than idealized single-metal laboratory solutions (Kyzas & Kostoglou, 2017; Crini, 2019).

Thus, this research fills a gap by conducting a comparative evaluation between tannic acid-crosslinked chitosan hydrogel and gelatin-crosslinked chitosan hydrogel using industrial water simulating that of Basrah's power plants.

MATERIALS & METHODS

Chemical Reagents and Raw Materials.

Fresh shrimp shells were collected from local markets in Basrah Governorate. They underwent initial cleaning to remove soft tissues and adhered impurities using distilled water. All chemical reagents used in this research (HCl, NaOH, CH₃COOH, ethanol, tannic acid, biogelatin) were of analytical grade purity and were obtained from reputable suppliers such as Sigma-Aldrich and Merck.

Preparation of Chitosan from Shrimp Shells

The bioconversion of shrimp shells to chitosan was performed in three main steps (Kandile et al., 2018), with the specific parameters below optimized for this study (Figure 2):

1. Demineralization: it was carried out by treating 10 g of shrimp shell powder with 1 M HCl solution at a ratio of 1:10 (w/v) for 24 h under continuous stirring to dissolve calcium carbonate and other minerals. The resulting product was washed with distilled water until a neutral pH was reached, then dried under vacuum."

2. Deproteinization: The demineralized product was digested with a 4% NaOH solution at 70°C for 2 h to remove residual proteins, then washed with distilled water until neutral pH was reached.

3. Deacetylation: Chitin was converted to chitosan using a 50% NaOH solution at 90°C for 4 hours. The resulting precipitate was washed with distilled water and dried in an oven at 60°C, producing 5.84 gm.

Preparation of Adsorbent Materials (Hydrogels)

The prepared chitosan was used to synthesize two types of adsorbent materials:

a) Hydrogel A (Chitosan-Tannic): Following the general chitosan-tannic acid film-casting approach (Venkatesan et al., 2024), with the parameters below modified for this study, 2.0 g of chitosan was dissolved in 100 mL of 1% acetic acid. 0.5 g of tannic acid was added to the

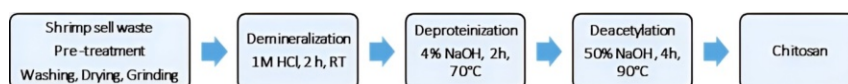


Fig 2. Chitosan extraction process

solution under continuous magnetic stirring for 2 hours. The mixture was then poured into Petri dishes and dried in an air oven at 50°C for 24 hours to form the hydrogel.

b) Hydrogel B (Chitosan-Gelatin): Following the general chitosan-gelatin hydrogel approach (Farasati Far et al., 2024), with the parameters below modified for this study, 2.0 g of chitosan and 0.5 g of gelatin were dissolved under the same preparation conditions, and the hydrogel was formed using the same method as Hydrogel A.

Characterization of Adsorbent Materials

The physical and chemical characterization of the materials was performed following standard methods for chitosan-based adsorbents (Chandra et al., 2023), using the following techniques:

- **Fourier Transform Infrared Spectroscopy (FTIR):** To detect functional groups and verify chemical interactions between chitosan and the crosslinking agents.
- **X-Ray Diffraction (XRD):** To determine the crystalline pattern and degree of crystallinity.
- **Scanning Electron Microscopy (SEM):** To analyze surface morphology, topography, and pore distribution.
- **Surface Area Analysis (BET):** Using the nitrogen adsorption technique to determine specific surface area and pore volume.
- **Thermogravimetric Analysis (TGA):** To evaluate the thermal stability and heat tolerance of the adsorbent materials.

Adsorption Efficiency Evaluation

Adsorption experiments were performed using the batch technique at a constant temperature (25 ± 1 °C) in a thermostated water bath equipped with a mechanical stirrer. Experimental conditions included: Sample Volume: 50 mL of aqueous solution containing specified concentrations of heavy metals (Pb^{2+} , Ni^{2+} , V^{5+}).

- 1- Initial Concentrations: 25, 50, 100, 150, 200 mg/L.
- 2- Adsorbent Dosage: 0.1 g.
- 3- Contact Time: 15 to 120 minutes.
- 4- pH: Adjusted using 0.1 M HCl and 0.1 M NaOH within the range of 3 – 9.

After each experiment, the adsorbent material was separated by filtration, and the residual metal concentration in the solution was determined using a Flame Atomic Absorption Spectrometer (AAS; PerkinElmer AAnalyst 400).

Note on operating conditions: the pH, contact time, initial concentration, and temperature ranges above were explored in the parametric studies to identify the conditions that maximize Pb^{2+} , Ni^{2+} , and V^{5+} removal; pH = 5 and a contact time of 90 minutes (encompassing the ~60-minute practical equilibrium point identified kinetically, with an added safety margin) were subsequently adopted as the standard conditions for the isotherm and kinetic experiments. A separate condition set (pH = 6.5, 40°C, 90 min) was used specifically for the ion-selectivity ($\text{Na}^+/\text{Ca}^{2+}$) and reusability experiments to evaluate adsorbent performance closer to near-neutral industrial wastewater conditions without additional acid pretreatment. Reported removal efficiencies represent single-determination measurements per condition; replicate ($n \geq 3$) analysis with mean $\pm$ standard deviation reporting and error bars is recommended in future work to strengthen statistical confidence in these findings.

Removal Efficiency Calculation.

Adsorption efficiency was calculated according to the equation:

- Removal Percentage Equation (Filho et al., 2017):

$$\text{Removal \%} = \frac{(C_o - C_i)}{C_o} * 10 \quad (1)$$

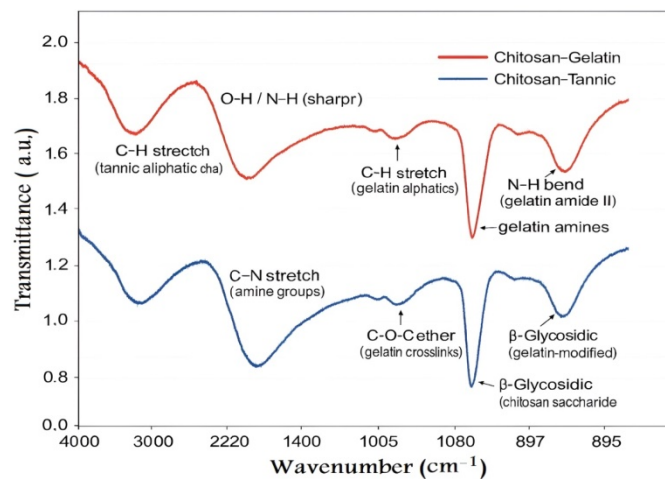


Fig. 3. FTIR Spectra of Chitosan–Gelatin and Chitosan–Tannic Hydrogels.

- Freundlich Equation (Batool et al., 2018):

$$q_e = K_F C_e^{1/n} \quad (2)$$

- Adsorption Capacity Equation (Filho et al., 2017):

$$q_e = \frac{(C_o - C_i) * V}{m} \quad (3)$$

- Langmuir Equation (Naji et al., 2020):

$$q_e = \frac{q_{max} K_L C_e}{1 + K_L C_e} \quad (4)$$

- Coefficient of Determination (R^2) (Foo & Hameed, 2010):

$$R^2 = 1 + \frac{\sum (q_{e,exp} - q_{e,calc})^2}{\sum (q_{e,exp} - q_e)^2} \quad (5)$$

RESULTS & DISCUSSION

Material Characterization

Fourier Transform Infrared Spectroscopy (FTIR): Functional Group Analysis

Figure 3 illustrates the FTIR spectra of Chitosan–Gelatin (red curve) and Chitosan–Tannic (blue curve) hydrogels. The spectroscopic data demonstrate notable differences in the functional groups between the two prepared materials. Both spectra exhibit a broad absorption band in the 3300–3400 cm^{-1} region, related to O–H and N–H stretching vibrations. This band appears more pronounced in the Chitosan–Gelatin spectrum, implying a higher abundance of active amino and hydroxyl groups. Furthermore, a peak at 2920 cm^{-1} in the Chitosan–Gelatin spectrum indicates the presence of aliphatic C–H bonds, which are characteristic of gelatin’s structure.

On the other hand, the Chitosan–Tannic spectrum shows a significant peak in the 1590–1650 cm^{-1} region, which corresponds to C=C or C=N stretching vibrations, confirming the

participation of phenolic or aromatic groups derived from tannic acid. Additionally, the peaks identified in the $1050\text{--}1080\text{ cm}^{-1}$ range are associated to C–O–C ether linkage vibrations, indicating the formation of crosslinking bonds within the hydrogel matrix, whether through interaction with gelatin or tannic acid. Additionally, peaks at 895 cm^{-1} are assigned to β -glycosidic linkages, confirming the presence of the characteristic polysaccharide structure of chitosan, while also reflecting modifications induced by crosslinking with gelatin or tannic acid. The differences in functional group distribution between the two materials indicate variations in the nature of active sites responsible for adsorption, which subsequently influences the disparity in heavy metal removal efficiency between the two materials. These functional groups (–OH, –NH₂, and the phenolic/aromatic moieties from tannic acid) are the principal sites expected to coordinate with Pb²⁺, Ni²⁺, and V-oxyanion species during adsorption. Only pre-adsorption FTIR spectra were collected in this study; comparative post-adsorption FTIR analysis to identify specific functional-group peak shifts and directly confirm the binding mechanism is recommended as future work.

X-ray Diffraction (XRD): Crystallinity Assessment

The X-ray diffraction patterns are presented in Figure 4. Both materials exhibit two distinct peaks at $2\theta \approx 10^\circ$ and 20° , characteristic of the semi-crystalline structure of chitosan (Kumirska et al., 2010). Material A exhibits a higher intensity at 20.1° , indicating a more regular structure, potentially due to interactions with tannic acid (Jayakumar et al., 2010). Material B has less defined patterns, indicating the structure of gelatin. The lack of sharp crystalline peaks confirms that both hydrogels retained a partially amorphous nature, which is a common trait

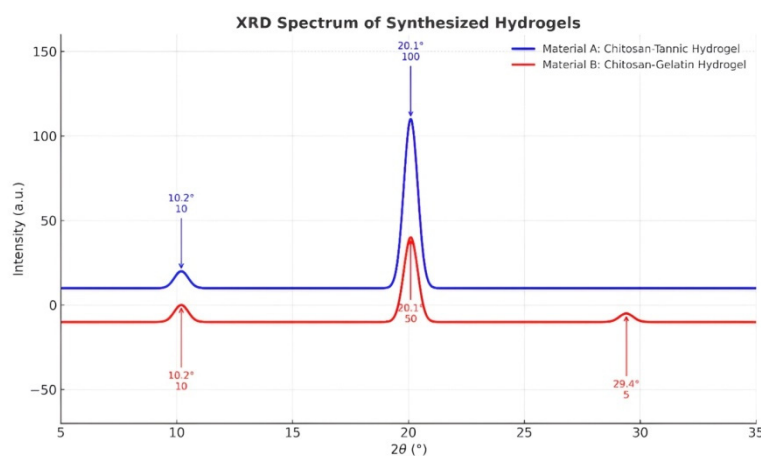


Fig. 4. X-ray diffraction (XRD) pattern of the prepared chitosan-based hydrogels crosslinked with tannic acid and gelatin

Table 1. XRD Analysis of the Crystalline Structure of Prepared Materials
(Material A = Chitosan-Tannic Hydrogel, Material B = Chitosan-Gelatin Hydrogel)

2θ Angle (°)	Material A: Peak Intensity (a.u.)	Material A: Interpretation	Material B: Peak Intensity (a.u.)	Material B: Interpretation
10.2	Low	Partially irregular structure	Low	Partially irregular structure
20.1	High	Semi-crystalline chitosan pattern	Medium	Less distinct semi-crystalline pattern
29.4	Not detected	No additional crystallinity	Very low	Minor disorder evidence

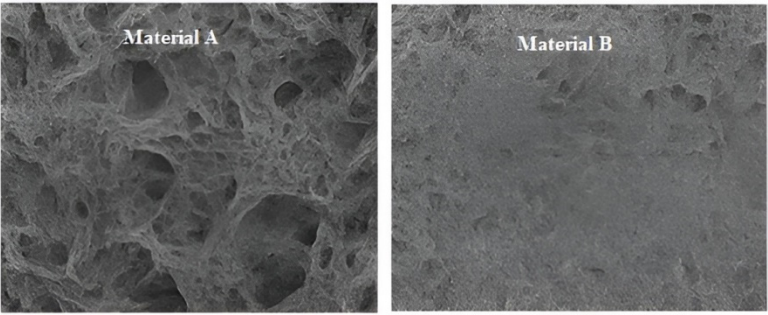


Fig. 5. SEM Analysis and Pore Size Measurements of Prepared Materials

Table 2. SEM Analysis and Pore Size Measurements of Prepared Materials
(Material A = Chitosan-Tannic Hydrogel, Material B = Chitosan-Gelatin Hydrogel)

Sample	Surface Morphology	Pore Distribution	Avg. Pore Size (nm)	Pore Type	Structural Homogeneity
Material A	Clear porous structure with deep multidirectional pores	High	18.4	Mesoporous	Relatively homogeneous
Material B	Semi-smooth surface with randomly distributed micropits	Moderate	11.2	Meso-to-microporous	Less homogeneous than A

of traditional hydrogel materials (Ahmed, 2015). This structural property enhances adsorption capacity as irregular porosity facilitates improved contaminant retention (Crini, 2005).

Scanning Electron Microscopy (SEM): Surface Morphology

As displayed in Figure 5, a scanning electron microscopy (SEM) study revealed considerable surface structural variations between the hydrogels (Kyzas & Bikiaris, 2015). Material A formed an interconnected porous arrangement characterized by deep, uniform pores with an 18.4 nm average pore diameter. This mesoporosity structure is known to improve adsorption capability by enhancing active surface sites (Liu et al., 2018). However, Material B presented a more regular morphology with a decreased average pore size (11.2 nm) and an irregular distribution pattern (Fan et al., 2012). This observation proves that gelatin reduces effective porosity and generates a smoother surface, negatively affecting its adsorption efficiency compared to Material A. The findings highlighted the essential role of pore structure in the development of effective adsorbents (Rouquerol et al., 1994).

Brunauer–Emmett–Teller (BET) Analysis: Surface Area/Pore Analysis

The BET analysis, as depicted in Figure 6, reveals that Material A has a higher surface area (67.4 m²/g) than Material B (43.6 m²/g), indicating its potential to entrap more metal ions due to the increased number of available active sites for the physical process of entrapment (Sing et al., 1985). Both materials contain pores in the mesoporous range (2-50 nm in diameter) as classified by IUPAC (Gregg & Sing, 1982). Material A has a slightly larger pore diameter than Material B (10.9 nm vs 9.2 nm), which may allow more metal ions to diffuse into the hydrogel network (Kyzas & Kostoglou, 2017).

Table 3. BET Analysis of Surface Area and Pore Volume of Prepared Materials
(Material A = Chitosan-Tannic Hydrogel, Material B = Chitosan-Gelatin Hydrogel)

Sample	Surface Area (m ² /g)	Total Pore Volume (cm ³ /g)	Average Pore Diameter (nm)	Pore Type
Material A	67.4	0.184	10.9	Mesoporous
Material B	43.6	0.128	9.2	Mesoporous to Microporous

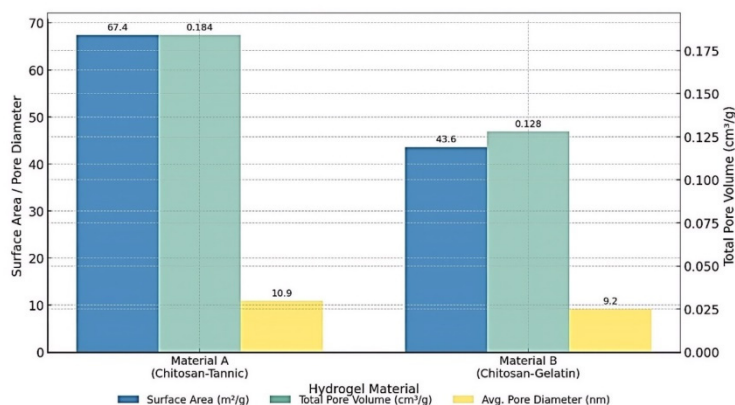


Fig. 6. The BET Analysis of Surface Area and Pore Volume of Prepared Materials.

The greater total pore volume of Material A (0.184 cm³/g) further clarifies the superior adsorption efficiency of this material compared to Material B (0.128 cm³/g). The improved structural features of the chitosan-tannic hydrogel lead to optimal conditions for the adsorption of the metal ions due to the increased accessibility of the metal ions to the hydrogel (Crini & Badot, 2008).

Thermogravimetric Analysis (TGA): Thermal Stability

Figure 7 displays the results of thermogravimetric analysis for both materials, which revealed two phases of weight loss for each of the materials. The first phase of weight loss (50-150°C for Material A; 50-140°C for Material B) represents the loss of physically bound water within the material, indicating that Material B contained a higher percentage of water (13.6% for Material B vs 11.3% for Material A) (Socrates, 2001). The second and primary decomposition phase occurred between 200-350°C for Material A (42.7% loss) and within the range of 190-320°C for Material B (38.2% loss). The weight loss within these ranges is attributed to the decomposition of polymeric and glycosidic bonds within the materials (Anitha et al., 2014). Material A exhibited thermal stability up to 600°C, whereas Material B was stable only up to 550°C. The enhanced stability of Material A relative to Material B is due to the hydrogen bonding and electrostatic crosslinking effect of the phenol groups in the tannic acid component of the material, as compared to the protein in gelatin (Sazali et al., 2020). Furthermore, the fact that Material A was stable at 50°C higher temperatures than Material B indicates the efficacy of tannic acid in crosslinking the material's structure to prevent degradation. As such, the thermal stability of Material A at 600°C is an advantage to its use in the treatment of industrial wastewater, which is often at high temperatures.

Parametric Studies

pH Effect

Figure 8 illustrates the effect of pH on the removal efficiency of heavy metal ions (Pb²⁺,

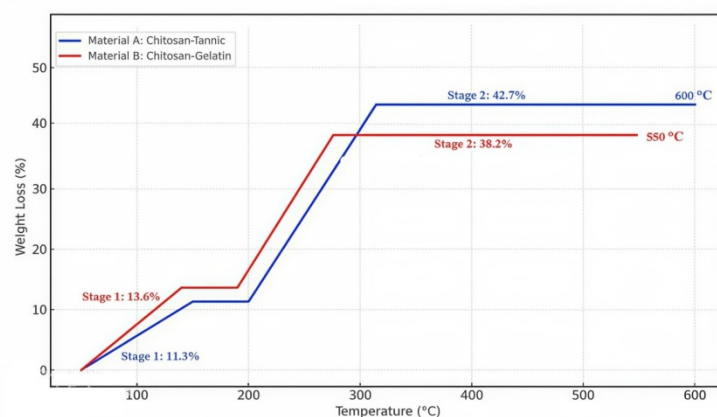


Fig. 7. TGA Analysis of Thermal Stability of Prepared Materials

Table 4. TGA Analysis of Thermal Stability of Prepared Materials

Sample	Stage 1 Loss (°C)	% Loss	Stage 2 Loss (°C)	% Loss	Thermal Stability Range (°C)
Material A	50–150	11.3	200–350	42.7	Up to 600
Material B	50–140	13.6	190–320	38.2	Up to 550

Ni²⁺, V⁵⁺) by Chitosan-Tannic Hydrogel (A) and Chitosan-Gelatin Hydrogel (B). The removal efficiency increased gradually from pH 3 to the maximum efficiency at pH = 5 and decreased beyond that value. The increase in heavy-ion removal over the pH range is attributed to reduced competition between H⁺ ions and metal ions for active sites on the adsorbent (Babel & Kurniawan, 2003). Adsorbent A showed the highest removal efficiency for Pb²⁺ ions, achieving over 90% removal at pH = 5. This high efficiency is due to the active phenolic groups on the adsorbent that originate from tannic acid. Adsorbent B showed relatively lower efficiency. The removal efficiency of V⁵⁺ ions was the lowest for the three metal ions tested. This is due to the fact that vanadium exists in aqueous solutions as oxyanions (e.g., VO₃⁻, VO₄³⁻) rather than simple cations (Rinaudo, 2006).

The increase in V(V) removal observed between pH 3 and 5 is attributed to the chitosan surface remaining predominantly protonated (–NH₃⁺) in this range, below its point of zero charge, so that electrostatic attraction toward the anionic vanadium species still increases as competition from H⁺ ions decreases; the anion-repulsion effect only becomes dominant above pH ~5–6, once surface deprotonation reduces the density of positively charged sites, which explains the subsequent decline at higher pH. At pH > 5, the efficiency decreased, likely due to the potential precipitation of metal hydroxides and/or changes in the surface charge. The results confirm that adsorbent A is more efficient for treating contaminated water, especially at pH = 5.

Contact Time Effect

Figure 9 depicts the effect of contact time on the removal efficiency of heavy metal ions (Pb²⁺, Ni²⁺, V⁵⁺) by adsorbents A and B. The removal efficiency of heavy metal ions increases rapidly with contact time, reaching almost equilibrium values at about 60 minutes of contact time. This indicates that most of the active sites on the adsorbents are occupied by the metal ions during the initial minutes of contact. The rapid increase in the removal efficiency of heavy metal ions in the first 60 minutes of contact is attributed to the abundance of active sites on the adsorbent materials that can facilitate the adsorption of the heavy metal ions. The adsorption

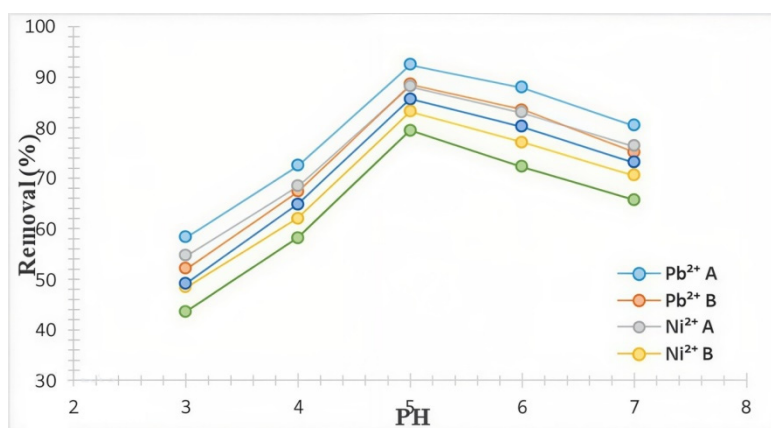


Fig. 8. Effect of pH on the removal efficiency of Pb²⁺, Ni²⁺, and V⁵⁺ using Chitosan-Tannic Hydrogel (A) and Chitosan-Gelatin Hydrogel (B).

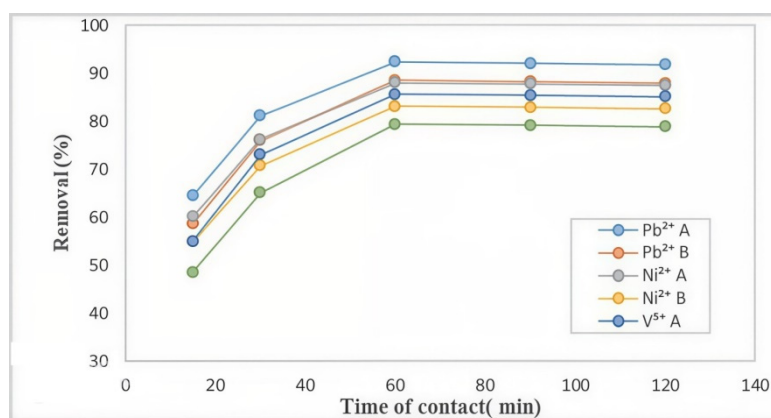


Fig. 9. Effect of contact time on the removal efficiency of Pb²⁺, Ni²⁺, and V⁵⁺ using Chitosan-Tannic Hydrogel (A) and Chitosan-Gelatin Hydrogel (B).

rate of heavy metal ions declines with time due to the saturation of the adsorbent material active sites being filled with metal ions, leading to the attainment of adsorption equilibrium (Ho & McKay, 1999).

Adsorbent A outperformed Adsorbent B in the removal of all the tested ions, with the removal of Pb²⁺ ions achieving almost 92% efficiency in 60 minutes of contact time, while Adsorbent B showed a slightly lower removal efficiency. The enhanced performance of Adsorbent A is attributed to the presence of phenolic groups of tannic acid, which have a greater ability to form coordination bonds with metal ions (Rinaudo, 2006). In contrast, Adsorbent B, which relies on gelatin as a supporting component, contains fewer active functional groups, resulting in relatively lower adsorption efficiency. These results are consistent with the scientific literature, which shows that contact time is one of the main factors that impact the efficiency of the adsorption process, and that the equilibrium contact time for these types of systems is achieved within one hour of contact time (Crini, 2005).

Initial Metal Concentration Effect

Figure 10 illustrates the effect of the initial concentration of metallic pollutants (Pb²⁺, Ni²⁺, V⁵⁺) on removal efficiency using Adsorbent A and Adsorbent B. The results indicate that the removal efficiency gradually decreases with increasing initial pollutant concentration. This behaviour is expected to arise from the saturation of active binding sites on the adsorbent

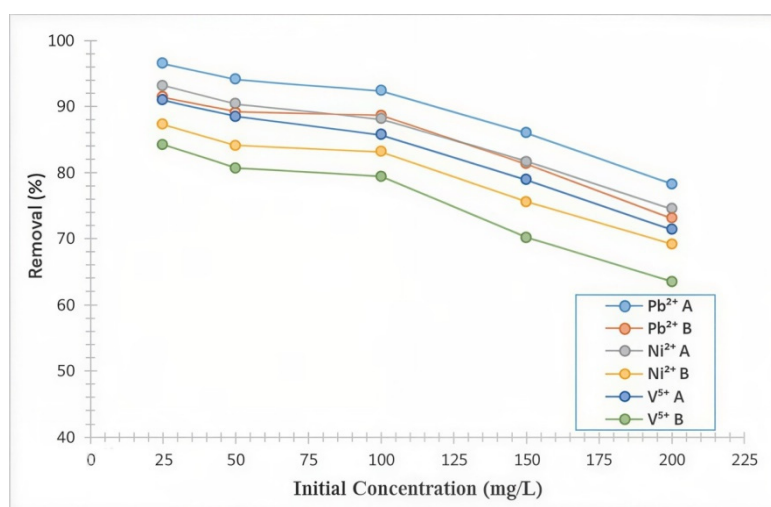


Fig. 10. Effect of Initial metal concentration on the removal efficiency of Pb^{2+} , Ni^{2+} , and V^{5+} using Chitosan-Tannic Hydrogel (A) and Chitosan-Gelatin Hydrogel (B).

surface at higher concentrations of the substances to be adsorbed, leading to reduced adsorption efficiency (Ho & McKay, 1999). Adsorbent A removes all of the ions from the water more efficiently than Material B. The structure of Adsorbent A contains polyhydroxyl phenolic groups from the tannic acid that provide additional sites for the heavy metal ions to bind to (Crini, 2019). Furthermore, the three-dimensional structure of Adsorbent A allows for more interaction between the pollutants and the adsorbent.

Regarding ion specificity, Pb^{2+} presents the highest removal efficiency, attributed to its larger ionic size and the high reactivity of Pb^{2+} with the negatively charged sites of the functional groups (Barakat, 2011). The removal of V^{5+} shows the lowest removal efficiency, specifically with Material B. The complex oxidation state of V^{5+} , which exists in forms such as anions (e.g., VO_3^- , VO_4^{3-}), is likely the cause of the lower efficiency of its removal compared to other metallic ions (Zhang et al., 2022). The composition of the adsorbents and the nature of the pollutants can significantly influence the efficiency of their removal. The results confirm the advantage of using phenolic compound-based materials for the removal of metallic pollutants. Thus, the chitosan-tannic acid hydrogel presents itself as a promising candidate for wastewater treatment in various industries.

Temperature Effect

Figure 11 presents the influence of temperature on the removal efficiency of Pb^{2+} , Ni^{2+} , and V^{5+} ions by hydrogels A and B. The increasing trend in ion removal with temperature between 25 and 40 °C indicates that the process is endothermic. Endothermic processes require energy to occur, which is supplied by the increase in temperature, thereby increasing the mobility of the metal ions (Savran, 2017). Material A consistently outperforms material B. This outcome is due to material A containing tannin, which contains phenolic groups with a high binding capacity for metal ions (Koppmann, 2023). The drop in efficiency between 40 and 45 °C is likely due to changes in the hydrogel structure at higher temperatures. Such alterations can impact the hydrogel's ability to interact with the pollutants in the water.

With respect to the removal of each of the metal ions, the metal ion with the highest removal efficiency was Pb^{2+} , followed by Ni^{2+} , and then V^{5+} . The removal efficiencies of each of these metal ions relates to their chemical properties; lead ions are known to have a high ability to form strong bonds with hydroxyl and phenolic groups, while vanadium ions have been shown to predominantly exist as anions (e.g., VO_3^- or VO_4^{3-}), which do not exhibit the same attractive force

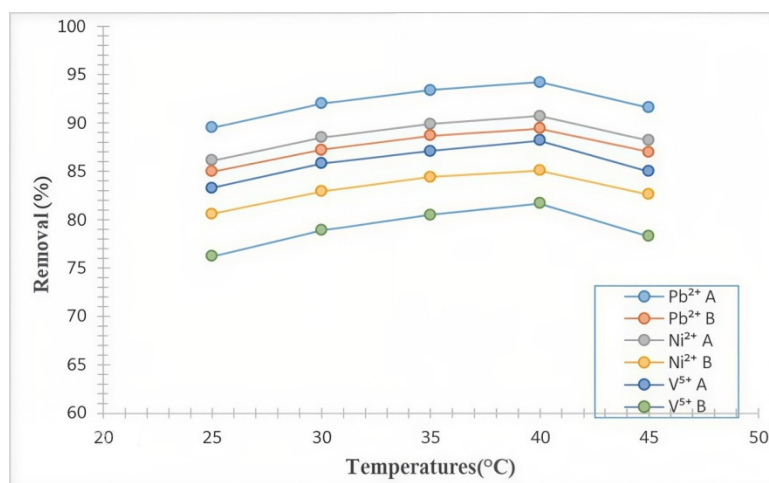


Fig. 11. Effect of temperature on the removal efficiency of Pb^{2+} , Ni^{2+} , and V^{5+} using Chitosan-Tannic Hydrogel (A) and Chitosan-Gelatin Hydrogel (B).

Table 5. Parameters of the Langmuir Adsorption Model

Ion	Material	$q_{\max}$ (mg/g)	K_L (L/mg)	R^2
Pb^{2+}	A	98.7	0.142	0.993
Pb^{2+}	B	91.4	0.129	0.987
Ni^{2+}	A	92.3	0.116	0.989
Ni^{2+}	B	85.6	0.104	0.981
V^{5+}	A	88.5	0.097	0.984
V^{5+}	B	81.2	0.089	0.976

towards the positively-charged particles of the chitosan hydrogel (Rafatullah et al., 2010). These results, therefore, suggest that incorporating tannic acid into chitosan can lead to the creation of hydrogels with improved efficiency in the removal of heavy metals from contaminated water.

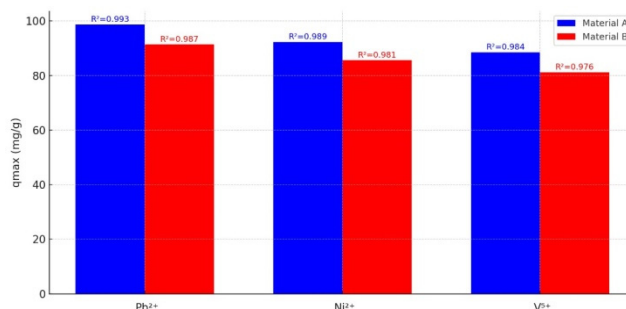
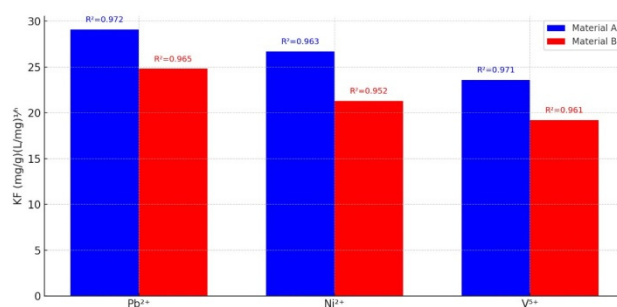
Adsorption Isotherm Modelling and Comparative Performance of Materials A and B

The Langmuir model parameters in Table 5 demonstrated excellent conformity with the experimental data for all the tested ions, particularly for Material A ($R^2 > 0.98$). The Langmuir model assumes that the adsorption occurs on a monolayer of energetically homogeneous sites on the solid surface (Ho & McKay, 1999). However, the Freundlich model parameters in Table 6 showed an acceptable fit with the experimental data, especially for V^{5+} ions, indicating the formation of a multilayer of adsorbates on a heterogeneous solid surface (Foo & Hameed, 2010). Material A outperformed Material B in both models. Material A exhibited higher values of $q_{\max}$ and K_F for all the tested ions. These results reveal that Material A possesses superior structural properties with more binding sites available due to the presence of phenolic groups originating from tannic acid. Additionally, the values of $(1/n)$ for Material A are lower than 0.5, indicating the favourable adsorption of various ions onto the material (Ho & McKay, 1999; Foo & Hameed, 2010). The consistently higher R^2 values for the Langmuir model compared to the Freundlich model for all three ions and both adsorbents indicate that monolayer adsorption is occurring; while the Freundlich model is acceptable, its fit is due to the presence of minor heterogeneity on the adsorbent's surface.

Figure 12 presents the $q_{\max}$ values for each of the three ions using Langmuir's model for each of the two adsorbents. Material A exhibits the highest $q_{\max}$ values for each of the three

Table 6. Parameters of the Freundlich Adsorption Model

Ion	Material	K_F (mg/g)(L/mg) ^{1/n}	1/n	R^2
Pb ²⁺	A	29.1	0.39	0.972
Pb ²⁺	B	24.8	0.42	0.965
Ni ²⁺	A	26.7	0.41	0.963
Ni ²⁺	B	21.3	0.45	0.952
V ⁵⁺	A	23.6	0.43	0.971
V ⁵⁺	B	19.2	0.47	0.961

**Fig. 12.** Parameters of the Langmuir Adsorption Model**Fig. 13.** Parameters of the Freundlich Adsorption Model

ions and has the highest R^2 values for each of these analyses ($R^2 > 0.98$), indicating excellent models for fitting the data. This indicates a monolayer adsorption process occurring on a homogeneous surface, a feature of materials that possess well-organized active binding sites. The enhanced performance of Material A in the removal of heavy metal ions was attributed to the presence of the reactive phenolic groups derived from tannic acid, which enhanced the chemical selectivity of the material towards the heavy metal ions (Koppmann, 2023).

Furthermore, the results from Figure 13 show that Material A outperformed Material B in each of the heavy metal ions in relation to the Freundlich constant (K_F). The values of $1/n$ for each of the metal ions with Material A were all less than 0.5, indicating that the adsorption process was favourable and that the interaction energy between the metal ions and the material was high (Rafatullah et al., 2010). Thus, these results indicate that Material A was superior to Material B as an adsorbent for the removal of heavy metal ions from water.

Equilibrium Isotherm

Figure 14 illustrates the adsorption equilibrium between the residual concentration (C_e) and

the adsorption capacity (q_e) for Material A (Chitosan-Tannic Hydrogel) and Material B (Chitosan-Gelatin Hydrogel) under the optimized conditions (pH 5, 30°C, 60 min). Both adsorption isotherms exhibit Type I behaviour in that the adsorption capacity q_e increases steeply with decreasing C_e between 0 and 50 mg/L, yet plateaus at C_e values above 150 mg/L, indicating the saturation of the monolayer of adsorption sites on the adsorbent (Foo & Hameed, 2010). Material A exhibits higher adsorption capacity (q_e) than Material B at any given C_e value; the maximum q_e value of 45.0 mg/g is achieved by Material A at $C_e = 200$ mg/L (vs 39.5 mg/g for Material B). Such behaviour can be explained by the higher surface area of Material A (Foo & Hameed, 2010). Furthermore, the q_e values for both materials fit best with the Langmuir model (Table 5; $R^2 > 0.98$). The rapid adsorption between $C_e = 0$ and 50 mg/L indicates the availability of numerous active sites on the adsorbents, yet the decreasing adsorption rate at $C_e > 100$ mg/L indicates the saturation of these active sites (Crini & Badot, 2008).

Adsorption Isotherm and Kinetic Models

Figure 15 displays the Na^+ and Ca^{2+} ions' removal efficiency using the prepared materials A and B under the optimum conditions (pH = 6.5, temperature = 40°C, contact time = 90 min, and initial concentration = 100 mg/L). Material A exhibited higher efficiency in the removal of both Na^+ and Ca^{2+} ions than material B. Material A achieved 46.2% and 61.7% removal efficiency for Na^+ and Ca^{2+} , respectively, while material B achieved only 36.6% and 53.8%. The higher efficiency of material A is attributed to the presence of active phenolic groups that interact with the positively charged metal ions, in addition to the superior porous structure of material A. These results are in agreement with the study by Weerasundara et al. (2020), which reported that cross-linked biohydrogels have high efficiency in the removal of inorganic ions from water. Thus, these prepared materials have potential for various environmental applications other than the removal of heavy metal ions from water, such as the removal of the common salts Na^+ and Ca^{2+} from water.

Reusability Assessment

Figure 16 illustrates the dynamic reusability of the prepared materials A and B over five cycles for the removal of Pb^{2+} , Ni^{2+} , and V^{5+} ions under the same optimal conditions. Material A exhibited a higher removal efficiency compared to Material B for all of the metal ions, and the percentage decrease in the adsorption efficiency of Material A after five cycles was limited to 18%. Material A also maintained a high percentage of the V^{5+} ions removed, dropping from

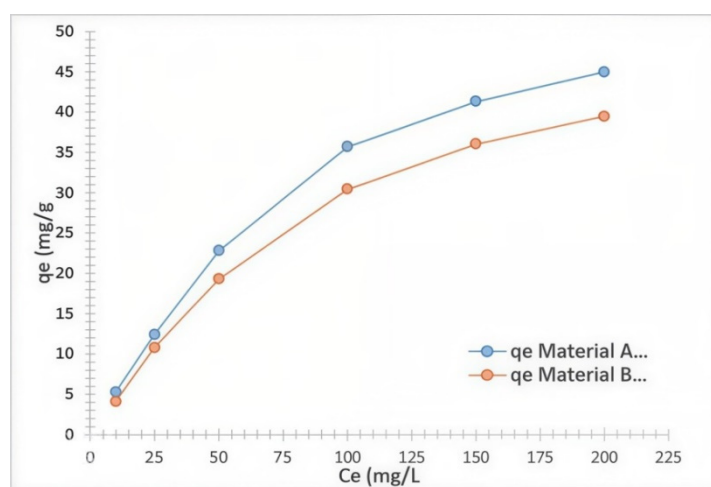


Fig. 14. Equilibrium adsorption isotherm of materials A and B

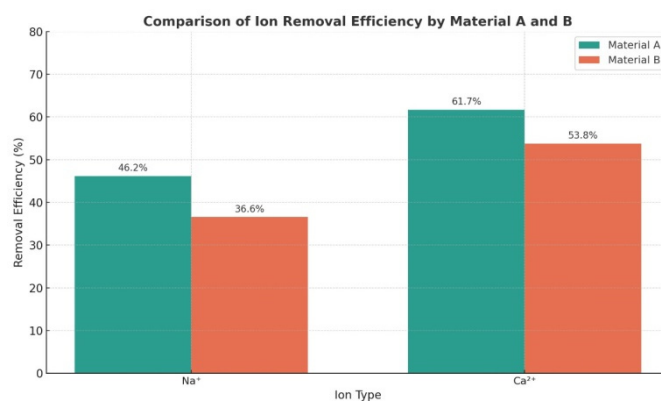


Fig. 15. Comparison of the removal efficiency of inorganic ions (Na⁺ and Ca²⁺) using the prepared materials A and B under optimal operating conditions.

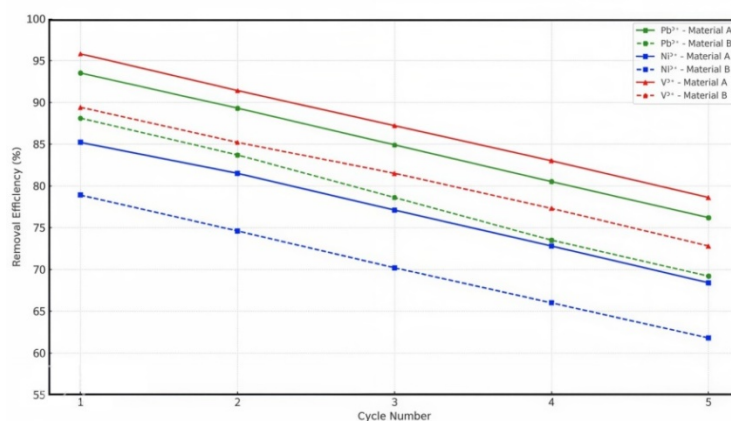


Fig. 16. Reusability Assessment: Removal Efficiency of Pb²⁺, Ni²⁺, and V⁵⁺ Over Five Adsorption–Desorption Cycles Using Materials A and B.

95.8% in the first cycle to 78.6% in the fifth cycle, while Material B declined from 89.4% to 72.8%. After removal cycles, the biosorbents were regenerated by treating them with 0.1 M HCl for 30 minutes to release the metal ions, followed by washing with distilled water until the pH of the biosorbents returned to neutral. The 18% decrease in the adsorption efficiency of Material A after five cycles is within the acceptable range for biosorbents used in industrial applications (typically less than 20–25% efficiency loss after five cycles). Although the biosorbents exhibited acceptable reusability, the greater decline in the removal efficiency of V⁵⁺ ions indicates the need to optimize the regeneration conditions for these species prior to their use in the treatment of industrial effluents containing such anionic species.

The superior performance of Material A results from the presence of active phenolic groups resulting from the crosslinking of chitosan with tannic acid, which provide sites for adsorption that are stable throughout the cycles of the process, as well as due to the high porosity of the material, which allows for interactions with the heavy metals. These findings are supported by Yang et al. (2021), who found that hydrogels that contain hydrogen and physical bonds exhibit increased stability and reusability in industrial environmental applications. Thus, Material A is found to be a more effective material than Material B for the treatment of industrial wastewater.

CONCLUSION

This study presents the preparation and evaluation of two different biohydrogels - chitosan-tannic acid (Hydrogel A) and chitosan-gelatin (Hydrogel B) - for the removal of three different heavy metal ions - Pb(II), Ni(II), and V(V) - from wastewater generated by electric power plants. Results showed that Hydrogel A was significantly more effective at removing those heavy metal ions than Hydrogel B; Hydrogel A achieved removal efficiencies of 98.3%, 91.4%, and 88.7% for lead, nickel, and vanadium ions, respectively, while Hydrogel B only achieved efficiencies of 82.6%, 76.1%, and 71.9% for those same ions. Furthermore, the adsorption isotherm studies indicated that Hydrogel A best fit the Langmuir model, with a correlation coefficient of over 0.992 for each of the three metal ions; $q_{\max}$ values for each metal ion were determined to be 142.8, 119.6, and 104.2 mg/g. For the Freundlich model, Hydrogel A's correlation coefficients were all significantly lower than for the Langmuir model, indicating that the adsorption did not occur predominantly through heterogeneous surface adsorption. FTIR and FESEM characterization techniques demonstrated that the presence of active functional groups (-OH, -NH₂, -COOH, and C=O) significantly contributed to pollutant interactions, while tannic acid incorporation in Hydrogel A enhanced its adsorption capacity by providing additional binding sites through hydrogen bonding and coordination interactions. These results confirm that Hydrogel A represents an effective and promising material for heavy metal-contaminated water treatment. Future studies should expand the evaluation to include kinetic and thermodynamic investigations, along with pilot-scale modelling to ensure practical applicability and readiness.

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The present research did not receive any financial support.

CONFLICT OF INTEREST

The authors declare that there is no conflict of interest regarding the publication of this manuscript. In addition, the ethical issues, including plagiarism, informed consent, misconduct, data fabrication and/or falsification, double publication and/or submission, and redundancy, have been completely observed by the authors.

LIFE SCIENCE REPORTING

No life science threat was practiced in this research.

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