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The environmental significance of *Citrullus lanatus* seed husk in biosorption of Pb^{2+}, Ni^{2+}, and Co^{2+} from municipal wastewaters

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Abstract

Heavy metal contamination in municipal wastewater poses a serious threat to aquatic ecosystems and human health. This study evaluated the potential of *Citrullus lanatus* seed husk biomass as a low-cost and sustainable biosorbent for removing lead (Pb^{2+}), nickel (Ni^{2+}), and cobalt (Co^{2+}) ions from wastewater. Batch experiments were conducted to optimize key operational parameters, including pH, temperature, retention time, biosorbent dosage, and particle size. Maximum biosorption efficiencies were achieved at pH near 7, 30–40 °C, and 30 min retention time, with a biosorbent dosage of 5 g/L and a particle size of 0.106 mm. Pb^{2+} exhibited the highest removal efficiency (up to 95.88%), followed by Ni^{2+} and Co^{2+}. Fourier transform infrared spectroscopy and scanning electron microscopy analyses confirmed the involvement of hydroxyl and carboxyl functional groups in the adsorption process. Biosorption behavior fits both the Langmuir and the Freundlich isotherm models, indicating favorable and heterogeneous adsorption. Municipal wastewater samples showed unacceptable heavy-metal levels after conventional treatment, whereas post-biosorption levels were reduced to below detectable limits. These findings highlight *C. lanatus* seed husk as an effective, sustainable, and economically feasible biosorbent for enhancing tertiary wastewater treatment and protecting aquatic ecosystems.

Keywords: *Citrullus lanatus*; Heavy metals; Biosorption; Wastewater; Adsorption kinetics; Adsorption isotherms

1. Introduction

It is widely recognized that technological advancements are often accompanied by increased rates of environmental pollution; these pollutants vary in form, chemical composition, hazard, and persistence.[1] Therefore, it has become imperative to develop safe methods to treat these pollutants and mitigate their toxic or harmful effects without disrupting the balance of any ecosystem.[2]

One of the most significant pollutants is toxic heavy metals, produced by human industrial activity or natural processes. Nevertheless, the danger of such pollutants stems from their ability to persist in the environment and accumulate in the tissues of living organisms, potentially reaching humans through food chains. Furthermore, they adversely affect organisms by accumulating in their tissues, leading to reduced reproduction and growth rates, increased mortality, and ultimately decreased biodiversity.[3] For example, chronic exposure to lead (Pb) could cause irreversible brain damage, kidney diseases, and behavioral issues in children, while nickel (Ni) and cobalt (Co) exposure could lead to allergies, asthma, lung fibrosis, and potential carcinogenicity.[4,5] Although the exact causes of autism are unclear, heavy metal contamination is widely suspected. Because of these risks, the United States Environmental Protection Agency (via the health guide of the Environmental Working Group) and the World Health Organization have established guideline values or standards for certain metals in water. Therefore, ongoing monitoring of these toxins is essential to maintaining a healthy and safe environment and, consequently, individual safety.[6] The petrochemical, fertilizer, textile, tanning, paint, and other diverse industries significantly contribute to the environmental burden of these metals. Hence, this pollution contributes to the deterioration of various ecosystems, which inevitably impacts human health. This critical type of pollution has created an urgent need for various treatment technologies, including adsorption, filtration, chemical precipitation, advanced oxidation processes, coagulation/flocculation, and biological treatment via microorganisms, which have been employed to treat such pollutants.[7] However, biological treatment, including biosorption, has recently attracted attention for its environmentally safe and sustainable properties and its potential to effectively treat industrial, municipal, and agricultural wastewater.[8] Recently, biosorption techniques have emerged as effective solutions for mitigating metal ions from various industrial effluents.[9] Plant wastes have played a significant role in this process because of their low cost, widespread availability, and excellent biosorption capacity.[10,11]

In Iraq, locals consume large quantities of watermelon (*Citrullus lanatus*), which is commonly consumed in cafes and at home as part of an ancient custom. Yet, it is also used in the production of local oils, jams, and soaps. Accordingly, these industries produce large quantities of discarded seeds. However, such plant-based residues, which are typically undesirable for animal nutrition, are often disposed of as municipal waste, thereby representing a sustainable source for water pollution treatment. Recent research has shown the potential of *C. lanatus* residues in the biosorption of heavy metal ions from various wastewaters. The biomass of this plant is widely available due to its widespread cultivation across Iraq, rapid growth, and ease of use in biosorption. Additionally, the cellular structure, chemical, and biological characteristics of this plant enhance its effectiveness in removing heavy metal ions, notably reducing their levels in various water systems. Therefore, *C. lanatus* is a promising candidate among plant-based bioremediation options.[12,13]

Finally, this study aims to improve the use of *C. lanatus* seeds in biosorption processes for wastewater contaminated with high levels of heavy metals, by studying various parameters that increase the efficiency of biosorption and understanding the mechanisms behind it, with the aim of developing the treatment of water contaminated with such pollutants using plant wastes.

2. Materials and methods

2.1. Preparation of biosorbent

C. lanatus seeds were collected from local fruit markets in Baghdad, Iraq. The seeds were thoroughly washed several times with tap water, then with distilled water, to remove contaminants and ensure purity. The seeds were then air-dried for 72 h.

To completely remove moisture and properly process the seeds, they were oven-dried at 90 °C for 24 h. The seeds were divided into two groups. The first group included seed biomass (SB) and pulp, whereas the second group included only seed husk biomass (SHB), as shown in Figure 1. The samples were ground and then sieved using a sieve with sizes of (0.106, 0.5, 1, and 1.5 mm). Grounded residues were stored in a dry atmosphere in tightly sealed glass bottles to maintain their integrity and efficiency in subsequent biosorption experiments.

2.2. Experiments of batch biosorption

The biosorption processes were investigated using various analytical methods, with a working volume of 100 mL of heavy metal solution kept in a 250 mL volumetric flask, at a concentration of 100 mg/L for Pb, Ni, and Co (Fluka, Switzerland). To optimize the experimental conditions of the biosorption process, the first experiment investigated the effect of the shape and type of *C. lanatus* biosorbents by comparing the biosorption of SB and SHB. Adsorbent powder (0.5 g) was added to each flask of 100 mL heavy metal solution, and the flasks were shaken at 120 rpm at 30°C for 60 min at neutral pH. The solution pH was adjusted using 0.1 M sodium hydroxide (NaOH) and/or hydrochloric acid (HCl). A control solution containing Pb^{2+}, Ni^{2+}, and Co^{2+} ions, without biosorbent, was prepared under identical environmental conditions to serve as a reference for comparison. After the biosorption process, all samples were centrifuged at 3,000 rpm for 40 min at room temperature, then filtered using a 45 μm membrane filter. The concentration of heavy metal ions in their aqueous solutions was measured using a flame atomic absorption spectrophotometer (AA-7000, Shimadzu, Japan). The group that demonstrated superior adsorption properties was then selected for all subsequent tests.

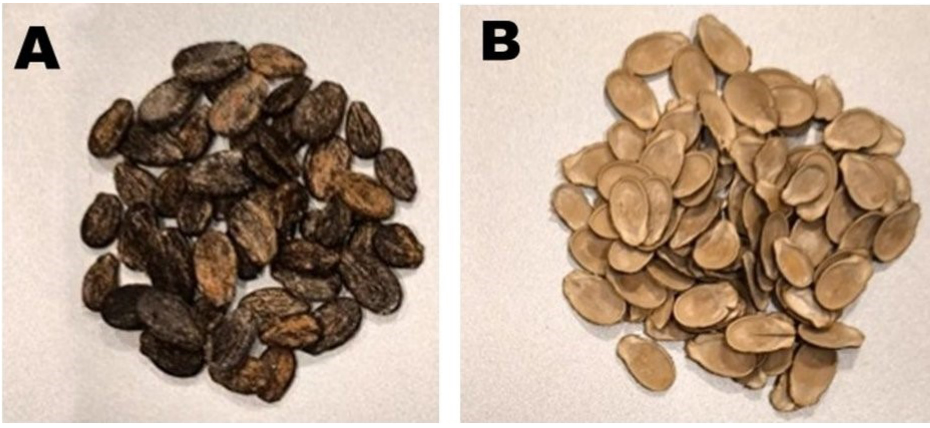

Figure 1. *Citrullus lanatus* seeds. (A) Seed biomass. (B) Seed husk biomass.

For further optimization of the biosorption processes, the effect of several key parameters was investigated, including pH in the range of 2–9, temperature at 30 °C for control circumstances and about 20–60 °C for other exploratory variables, retention time in the range of 5–120 min, biosorbent concentration of 1, 2.5, 5, 10, and 20 g/L, and the powder particle size of 0.106–1.5 mm. Nevertheless, to study the effect of particle size on overall biosorption efficiency, the initial biosorbent concentration was 10 g/L. The optimal pH was selected for subsequent biosorption experiments. The abovementioned procedures were applied consistently and uniformly in all subsequent experiments to determine the optimal efficient biosorption process. The overall efficiency of the biosorption process and the adsorbed quantity at equilibrium (*qe*) in mg/g were measured using **Equations 1** and **2**:

$$\text{Biosorption effeciency }\% = \frac{Ci - Cf}{Ci} \times 100 \quad (1)$$

$$qe = (Ci - Cf) \times \frac{V}{W} \quad (2)$$

where (C*i*) and (C*f*) are the initial and final concentrations of heavy metal, respectively. *V* (L) represents the volume of the heavy metal solution, while *W* (g) represents the weight of the biosorbent. To ensure the accuracy of the results, all measurements were performed in triplicate, and mean values were reported.

2.3. Determination of *Citrullus lanatus* seeds pH at zero charge and scanning electron microscopy and Fourier transform infrared spectroscopy analyses

The titration procedure was applied to detect the pH at zero charge (pHZC) of *C. lanatus* SHB. To perform that, a series of sodium chloride solutions (0.1 M) was prepared in 100 mL flasks. The initial pH (pH*i*) of each solution was adjusted to 2–13 using 0.1 M NaOH or HCl and a pH meter (Orion Star A211, Thermo Scientific, Indonesia). The final volume was fixed at 25 mL for each solution. Afterward, 0.5 g of sample powder was added to each solution, which was then shaken at 120 rpm for 24 h. The final pH (pH*f*) was recorded. The difference between pH*i* and pH*f* (ΔpH = pH*i* − pH*f*) was plotted against pH*i*, and pHZC was determined as the point where ΔpH equals zero on the *x*-axis.

The scanning electron microscopy (SEM; Vega III, Tescan, Czech Republic) was employed to identify the morphology of surfaces for both metal-treated and raw SHB, where a mixture of Pb, Ni, and Co metals solution was prepared at a concentration similar to the initial concentration used in all previous experiments (100 mg/L) and under the same treatment conditions (1 g of adsorbent), in order to prepare a sample for electron microscopy that mimics the initial experiments for all variables. Fourier transform infrared spectroscopy (MB3000, ABB Spectrolab, UK) was used to investigate the functional groups responsible for metal adsorption in both unsaturated and saturated biosorbents. To implement this test, the samples were dehydrated, combined with potassium bromide (KBr) (Fluka, Switzerland) at a 1:10 ratio, ground, and pressed into pellets. After subtracting the KBr background, all spectra were displayed on the same absorbance scale.

2.4. Biosorption isotherms

The Langmuir and the Freundlich isotherm models were used to fit the experimental results and analyze the adsorption behavior of heavy metal ions onto the biosorbent.[14,15]

The linearized form of the Langmuir equation is shown in **Equation 3** below:

$$\frac{1}{qe} = \frac{1}{KL\,qmax} \times \frac{1}{Ce} + \frac{1}{qmax} \quad (3)$$

where ($qmax$) and (KL) are constants of Langmuir, representing the maximum capacity of adsorption and adsorption energy, respectively. Ce and qe are the equilibrium concentration (mg/L) and quantity adsorbed at equilibrium time (mg/g), respectively.[16,17]

Alternatively, the Langmuir adsorption isotherm can be represented using the equilibrium parameter (RL), which is a dimensionless constant separation factor (**Equation 4**):

$$RL = \frac{1}{1 + KL \times Ci} \quad (4)$$

where Ci refers to the initial concentration (mg/L), and KL is the Langmuir constant.[18]

The Freundlich isotherm model defines adsorption as a phenomenon that occurs over a heterogeneous surface via a multilayer adsorption process,[19] as shown in **Equation 5**:

$$Inqe = InKf + \frac{1}{n} \times InCe \quad (5)$$

where Kf and n are Freundlich constants related to bonding energy. The adsorption coefficient Kf indicates the amount of metal ions adsorbed onto the adsorbent given a unit equilibrium concentration. The adsorption intensity of metal ions onto the adsorbent is indicated by $1/n$, with the adsorbent regarded as more heterogeneous as $1/n$ approaches zero.[20]

2.5. Municipal wastewater collection samples

Wastewater samples (before treatment) were colcted from the main basin of Rustumiya sewage treatment plant (RSTP) and from Diyala River (after treatment). RSTP is located in southern Baghdad, Iraq, within a heavily guarded military area (global positioning system coordinates: 33°17'19.3" N, 44°31'50.7" E). This plant has been one of the largest municipal wastewater treatment plants since 1963, as all municipal wastewater sources in the Rusafa side of Baghdad flow into it. This water contains a high concentration of pollutants and is treated through various processes, including primary treatment (screening and skimming), and secondary treatment (sedimentation tanks, aerobic oxidation tanks, and activated sludge tanks). The treated water is discharged into the Tigris River via the Diyala River. This plant does not include tertiary treatment processes. Sewage samples were collected using sterile, dark colored, 250 mL plastic bottles with tight caps. Samples were taken to the laboratory for chemical analyses, including measurements of Pb^{2+}, Ni^{2+}, and Co^{2+}, with biosorption conditions applied to samples from Diyala River after RSTP treatment, in accordance with lab samples that underwent *C. lanatus* biosorbent.

2.6. Statistical analysis

The one-way analysis of variance (ANOVA) test was used to analyze all experimental data. The Statistical Package for Social Sciences (SPSS 29.0, IBM, United States) was used for statistical analyses. All computations, regression analyses, and graphs for the Langmuir and the Freundlich models were prepared using Origin software (V.8, OriginLab Corporation, USA).

3. Results and discussion

The present study investigates the effects of several environmental parameters on the efficiency of the biosorption process using *C. lanatus* seeds for the removal of heavy metal ions at a constant and uniform initial concentration of 100 mg/L, to determine the optimal conditions for maximum removal of these hazardous pollutants.

3.1. Biosorption using *Citrullus lanatus* seed biomass and seed husk biomass

Both *C. lanatus* SB and SHB have the potential to perform biosorption for pollutant removal, but their efficiency and mechanism differ due to differences in composition and structure.[21] The pH was maintained at 7 throughout the experiment to ensure a clear, pure negative charge on the adsorbent surfaces, thereby increasing affinity for positively charged metal ions via electrostatic interactions. The pH was selected because pH 7 is a critical value; above 7, heavy metal ions precipitate, causing interference between the adsorption and precipitation processes. Preliminary results indicate superior biosorption efficiency with SHB compared to SB. SHB's biosorption efficacy (93.6 ± 2.8%, 70.2 ± 1.8%, 56.9 ± 1.04% for Pb^{2+}, Ni^{2+}, and Co^{2+}, respectively) was found to be higher than those observed for SB, which recorded 74 ± 1.9% for Pb^{2+}, 42.1 ± 0.8% for Ni^{2+}, and 31.7 ± 0.6% for Co^{2+}, as shown in Figure 2. Statistical analysis of the results showed significant differences ($p < 0.001$) in the efficiency of biosorption for both biomass forms of the studied heavy metals. Furthermore, post hoc comparisons using the least significant difference (LSD) test ($\alpha = 0.05$) revealed that SHB outperformed SB in removing heavy metal ions, with an LSD value of 2.854, supporting the use of this biomass, especially since it can be easily obtained at low cost from local cafes and factories that produce such waste.

The biosorption efficiency using SHB followed the sequence: Pb > Ni > Co, indicating different binding affinities for the studied heavy metals. SB, including the

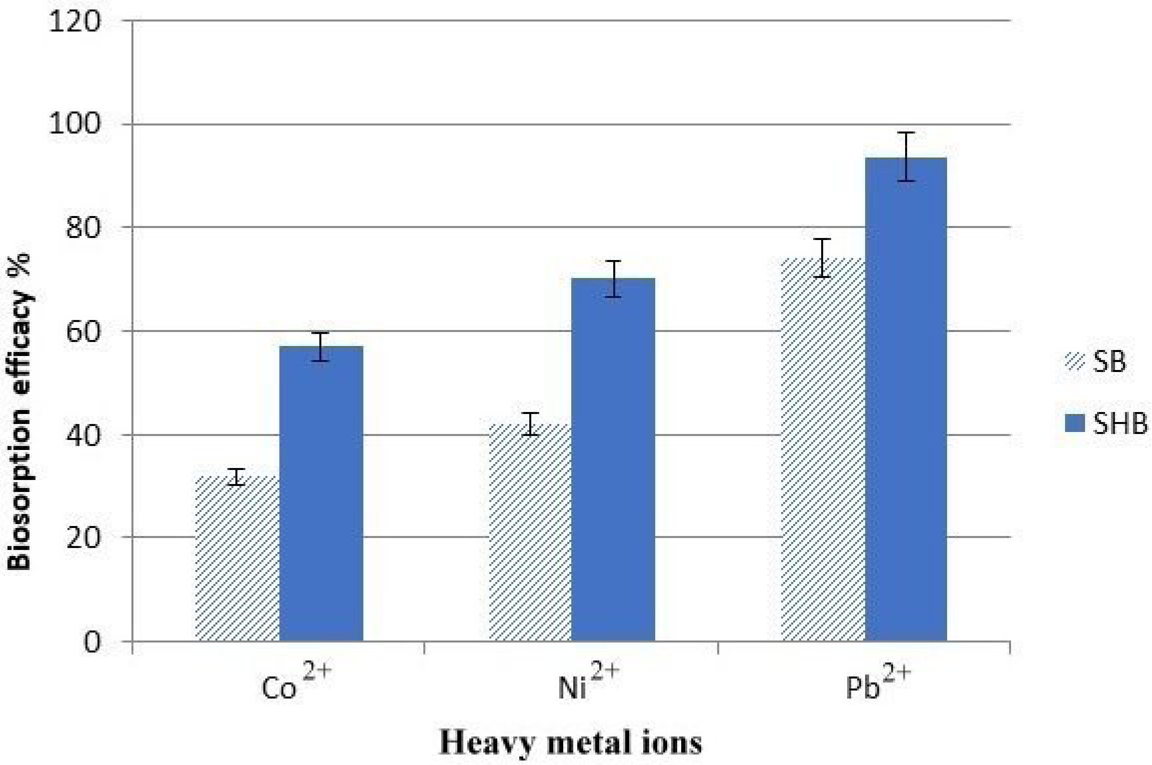

Figure 2. Biosorption differences between *Citrullus lanatus* seed biomass and seed husk biomass on heavy metal ions

core, contains 22% protein, 2.5% ash, 39% fiber, 11% carbohydrates, and 21% vegetable oil (saturated and unsaturated fatty acids), in addition to various simple compounds that consitute the remaining percentage.[22] To improve the efficiency of the biosorption process, these seeds (SB) must be modified.[23] Meanwhile, SHB consists of a high percentage of cellulosic fibers, pectin, and various polymers, all of which are rich in active carboxyl and hydroxyl groups that can bind heavy metal ions easily, quickly, and effectively through the electrostatic attraction mechanism.[24] Hence, these SHB do not require any modifications that may be economically unfeasible, as they can be used directly in the treatment process. Therefore, SHB was selected for subsequent biosorption experiments in this study.

3.2. pH zero charge

The pHZC is an important parameter, as it provides preliminary insight into the surface charge characteristics of the adsorbent used in the biosorption process and its behavior. The present study revealed that the pHZC for SHB was 6.6 (Figure 3), indicating a negative surface charge at pH values above 6.6 and a positive surface charge at pH values below 6.6. This result is crucial because it reveals the optimal pH range for the biosorption process, which depends on the affinity between heavy metal ions and the adsorbent surface charges.[25] Compared to other plant-derived biomasses, the pHZC value of peanut shells was found to be 6.7,[26] while it was 6.53 in the case of orange peel.[27]

Generally, high biosorptive elimination of cationic heavy metals is achieved at a high pH solution, the biosorbent surfaces are negatively charged (alkaline conditions), while they are the opposite for anionic heavy metal forms, where lower pH values or strong acidic conditions lead to functional group protonation that increases the binding capacity of such surfaces to anionic heavy metals.[28] The current results indicate that pH is a key factor in assessing optimal biosorption performance.

3.3. Biosorption characteristics optimization

3.3.1. Effect of pH

The biosorption efficiency of *C. lanatus* biomass was clearly affected by pH values, as shown in Figure 4. The best performance was achieved between pH 6 and 7, around pHZC of 6.6. Statistical analysis ($p < 0.001$) showed

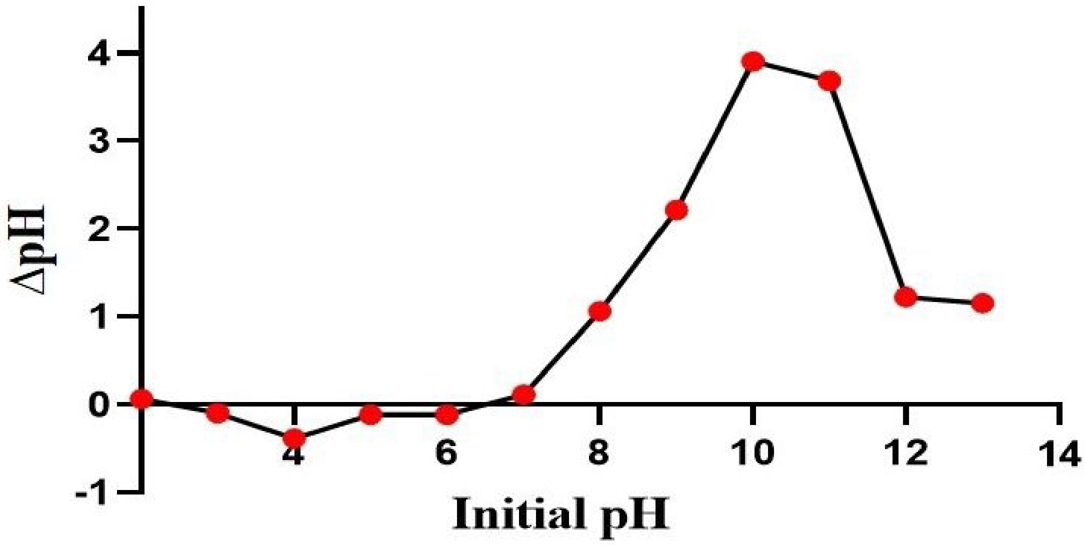

Figure 3. pH zero charge of *Citrullus lanatus* seed husk biomass at 30 °C, 120 rpm, pH range 2–13

significant differences in the uptake of heavy metal ions across pH values. At pH values below pHZC, the positively charged surface of SHB repelled metal cations, reducing the efficiency of biosorption. As pH values increased above pHZC, the SHB surfaces became negatively charged, strengthening electrostatic attractions and consequently increasing ion removal and improving treatment efficiency.

The heavy metal ion with the greatest biosorption percentage was Pb^{2+} (95.48%) at pH 6, which may be due to the intense affinity of this metal for carboxyl and hydroxyl active groups on the biomass surfaces. On the other hand, SHB showed lower adsorption efficiencies toward Ni^{2+} and Co^{2+} (70.22% and 58.4%, respectively) at pH below 6, because of differences in binding affinity between the biomass functional groups and the individual metal ions. Intriguingly, a marginal drop in biosorption activity was noticed at pH 7 for all studied metal ions, possibly due to a slight precipitation effect.[23-29]

The results indicate the crucial role of pH in affecting the surface charges of SHB and, consequently, the biosorption process, identifying pH near 7 as the optimum value for achieving the highest uptake efficiency. This is in agreement with the findings of a prior study by Lakshmipathy and Sarada,[30] who reported that *C. lanatus* plant residues achieved the highest biosorption of heavy metal ions at near-neutral pH, with minimal precipitation effects. Likewise, another study showed that the highest remediation efficiency of heavy metals by *C. lanatus* was at pH 7, and this removal was attributed to adsorption rather than precipitation.[31] These findings are consistent with the findings of the current study, which confirms that the optimum remediation process was near-neutral pH using SHB. Cellulose and various polymers within the SHB structure contain a large proportion of active hydroxyl and carboxyl groups that have a clear role in binding to heavy metal ions.[13-32] As pH values rise, these active groups deprotonate and enhance their capability for interacting and binding with the metal cations through complex formation and electrostatic attractions. Therefore, adsorbents' ability to interact with the relevant cations may be assessed by their cation-exchange capacity at a specific pH.

Biosorption is considered a complex process involving other mechanisms such as complex formation, physical adsorption, and precipitation.[33] In general, polysaccharides such as cellulose, as well as structurally similar biopolymers, are widely used in water management processes worldwide due to their capability to adsorb a variety of pollutants, including dyes, heavy metals, pesticides, phenols, and detergents. For numerous heavy metal ions, biosorption efficacy increases at neutral to alkaline pHs, typically ranging from 6 to 8, owing to reduced competition with hydrogen ions and enhanced electrostatic attraction between metal cations and the adsorbent.[23-34] However, the favorable pH range varies depending on the variety of heavy metals and the adsorbent used, as several heavy metal ions show different binding affinities depending on the pH.

3.3.2. Effect of temperature and retention time

To assess the effect of temperature on the biosorption efficacy of the studied heavy metals (Pb^{2+}, Ni^{2+}, Co^{2+}), a

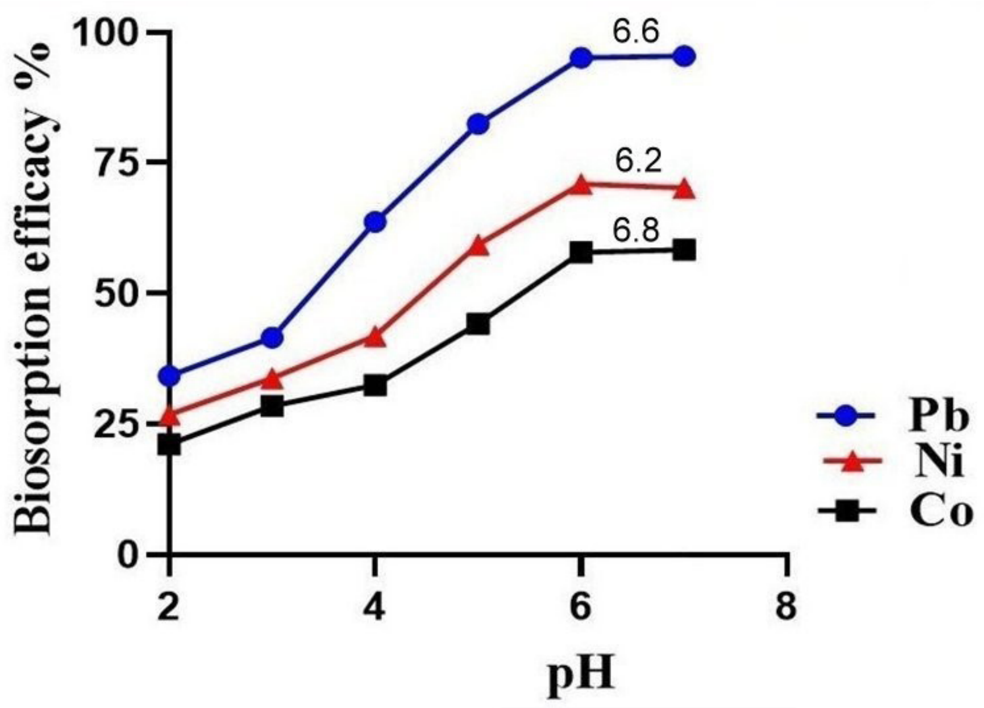

Figure 4. Effect of pH on biosorption using *Citrullus lanatus* seed husk biomass

temperature range from 20 °C to 60 °C was studied. The results showed a clear relationship between the efficiency of the biosorption process and the change in temperature, through a significant increase in the efficiency of the biosorption process with increasing temperature to the optimum temperature range of (30–40 °C), where the efficiency decreased after this temperature range (Figure 5A). At low temperature (20 °C), all examined metals had poor biosorption effectiveness rates. Biosorption efficiency of Pb^{2+} achieved the highest at 30–40 °C and decreased slightly after 50 °C. The other metals showed similar temperature patterns but with lower biosorption rates (Pb > Ni > Co). The observed biosorption behavior indicates that temperature significantly influences the thermodynamics of the biosorption process, with a significant positive enthalpy change ($^{+}\Delta H°$), suggesting an enthalpy-dependent process. However, the initial increase in the efficiency of biosorption is typically caused by the enhanced kinetics of heavy metal ions and interactions with the SHB, while the decrease in biosorption efficiency at temperatures above the optimum may be due to saturation of the active binding sites, decomposition of the SHB, or a change in the oxidation state of the metal. Our findings align with those of earlier studies, indicating the critical role of temperature in achieving optimal conditions for the biosorption process to obtain the highest removal rate of heavy metal ions.[35] The importance of the current study lies in SHB's ability to achieve the highest biosorption efficiency at 30 °C. This enables an optimal temperature with effective, practical economic feasibility compared to other plant biomasses that require higher temperatures to achieve acceptable adsorption efficiency, sometimes up to 50 °C.[36] This can reduce energy and operating costs, thereby enhancing and supporting the possibility of field application of the process on a larger scale.

To further optimize biosorption conditions, the effect of retention time on biosorption performance was studied. The obtained results revealed a gradual increase in the adsorption rate of metal ions over time, reaching the highest removal rate at 30 min (Figure 5B). At the beginning of the experiment, a 5-min retention time showed low biosorption efficiencies for all heavy metals (Pb^{2+}, Ni^{2+}, and Co^{2+}) of 73.41%, 59.83%, and 44.33%, respectively. However, as retention time increased, a marked improvement in biosorption was observed, peaking at 30 min. After this period, relative stability of the biosorption process was observed, with a slight variation in removal efficiency at 60–120 min, indicating that the adsorption process achieved equilibrium within 30–60 min.

In general, the results indicate that the optimum conditions to achieve the highest elimination efficiency for the heavy metal ions were 30 °C for 30 min. This condition

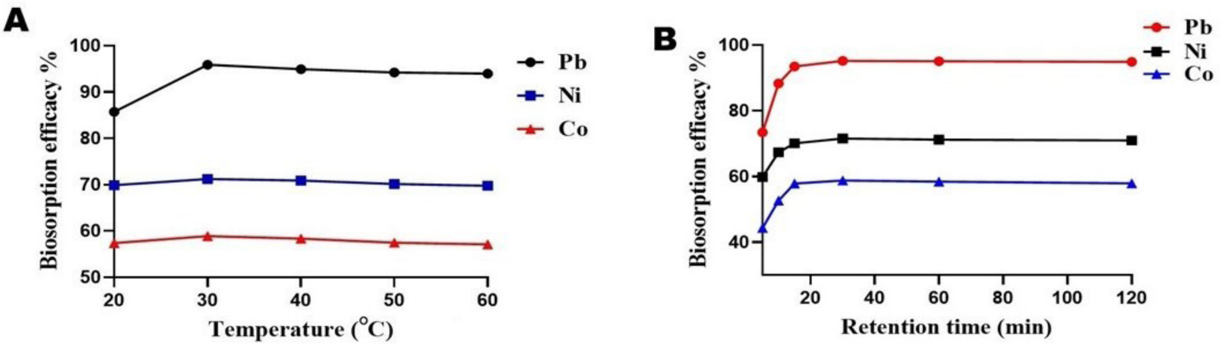

Figure 5. Effect of (A) temperature and (B) retention time on biosorption efficacy of *Citrullus lanatus* seed husk biomass

helps reduce operating costs and energy consumption while ensuring optimal removal of metal ions.

3.3.3. Effect of husk powder size and weight

Recognizing the impact of SHB size and weight on the biosorption process is essential to improve the removal efficacy of metal ions, as these variables directly affect access to active binding sites and, thus, the overall performance of the adsorbent biomass. Figure 6A illustrates that the efficiency of the biosorption process for Pb^{2+}, Ni^{2+}, and Co^{2+} using SHB is highly dependent on the concentration of the adsorbent, with an optimum biosorbent concentration of 5 g/L for the studied metal ions. The efficiency of the biosorption decreased beyond this concentration due to the saturation of the active binding sites with the associated metal ions. Related effects of saturation were observed in previous studies using various agricultural wastes to adsorb heavy metals, demonstrating the significant impact of binding-site saturation on the overall treatment process.[12] The results from our study indicate that SHB exhibited the highest biosorption efficiency toward Pb^{2+} (95.38%), consistent with its strong affinity for the hydroxyl and carboxyl active binding groups within SHB. In comparison, SHB demonstrated lower biosorption efficiencies toward both Ni^{2+} and Co^{2+} (71.13% and 58.88%, respectively), at the same concentration of 5 g/L, due to their weak binding affinity.[37] These results confirm the potential of SHB as a low-cost, environmentally friendly, and sustainable biosorbent for heavy metal removal, while highlighting the need to optimize the biosorbent weight to avoid saturation, which reduces the treatment efficiency.

Current results show that the particle size of SHB powder had a significant and critical effect on the efficiency of the biosorption process (Figure 6B). Small sizes (e.g., 0.106 mm) exhibited the highest biosorption efficiency, while this efficiency decreased at larger sizes (e.g., 1 mm and 1.5 mm) due to the decrease in the surface area of the particles and limited access to the active binding sites. This is consistent with the findings of a previous study by Sharma and Devi,[38] who reported that the small particle size of snail shell powder increased the efficiency of metal ion removal from water, confirming the importance of available surface area and active binding sites. In the same context, a study of the biosorption efficiency of prickly pear cactus and agave plant fibers showed that they have a high ability to remove Pb^{2+} ions, reaching 93% at a particle size of 572 μm.[39]

Overall, our study highlights that the biosorption capacity can be enhanced by introducing additional binding sites for metal ions. Furthermore, optimization of concentration and particle size is necessary to increase activity without reaching saturation. The ANOVA test detected highly significant differences in the biosorption of heavy metals with SHB powder size ($p < 0.001$), affecting the biosorption process for all investigated metals. Post hoc comparisons using the LSD test ($\alpha = 0.05$) indicated that mean differences exceeding 0.801 were statistically significant. In addition, the concentration of the biosorbent had a significant effect on heavy metal biosorption (ANOVA, $p < 0.001$), with an LSD value of 2.21 at the 5% significance level.

3.3.4. Fourier transform infrared spectroscopy and scanning electron microscopy analysis

After treatment, a broad peak at 3,439 cm^{-1} corresponded to –OH stretching vibrations, showing the existence of surface hydroxyl groups. This peak shifted slightly to 3,462 cm^{-1} and became broader and more intense, confirming the presence of a related –OH group with the element. The peak at 2,922 cm^{-1} was attributed to C–H stretching of aliphatic $-CH_2$/$-CH_3$ groups. It shifted slightly to 2,945 cm^{-1}, indicating a probable structural rearrangement or the introduction of additional aliphatic groups due to treatment. Peaks at 1,742 cm^{-1} and 1,642 cm^{-1} indicated

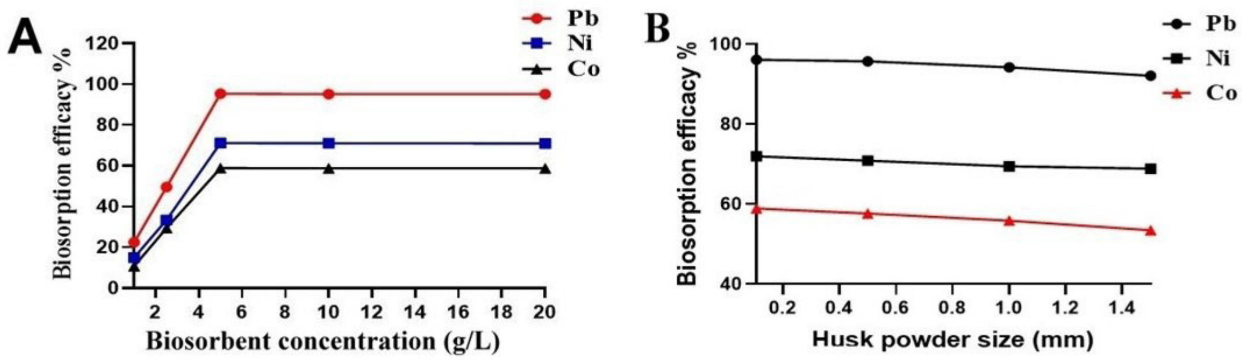

Figure 6. The effects of (A) concentration and (B) powder size on the biosorption efficacy of *Citrullus lanatus* seed husk biomass

C=O stretching of carbonyl groups. The bands shifted to 1,752 cm^{-1} and 1,626 cm^{-1}, respectively, with slight intensity changes, indicating the presence of a carbonyl functional group, potentially due to the adsorption of new species during treatment. A peak at 1,449 cm^{-1} suggests C=C stretching; this band shifted to 1,379 cm^{-1}, indicating a breakdown of conjugated structures following treatment. Additionally, the C–O stretching band at 1,048 cm^{-1} shifted to 1,063 cm^{-1}, indicating that oxygen-containing functional groups altered after treatment. Peaks at 623 cm^{-1} and 508 cm^{-1} may suggest C–H bending, which occurs when elements interact with carbon molecules following treatment. Heavy metal ion interactions during treatment generate new and shifted peaks at 901 cm^{-1}, 646 cm^{-1}, and 515 cm^{-1}, indicating alterations in the aromatic or heterocyclic structure (Figure 7).

To investigate the changes that may occur in the SHB surface morphology due to biosorption, SEM imaging of SHB before and after biosorption treatment was used. A synthetic solution containing 100 ppm of Pb^{2+}, Ni^{2+}, and Co^{2+} was used. Figure 8 shows SHB before biosorption at 1,000 × and 5,000 × magnifications, and SHB after biosorption of Pb^{2+}, Ni^{2+}, and Co^{2+} ions at 1,000 × and 5,000 × magnifications. Initial SEM observations showed that SHB exhibited light- and dark-colored areas. The light-colored areas contain inorganic elements, such as phosphorus and sodium, while the dark areas represent organic proteins and carbon-rich cellulose fibers.[22] Figure 8C also shows a wide distribution between small and large particles (appearing on smooth surfaces), with small-sized objects predominating, at approximately 10 µm. While Figure 8B reveals the presence of clear clusters of small objects and less distribution between small and large particles, in addition to the presence of more clear fibrous structures with relatively rough surfaces (Figure 8D), as an indication of the occurrence of the biosorption process. It is noted that the surface of the SHB shows light-colored crusty deposits due to the presence of heavy metals after treatment.

3.4. Adsorption isotherm

3.4.1. Langmuir adsorption isotherm

The Langmuir isotherm model may be used to describe adsorption equilibrium and to generate parameters ($qmax$ and KL) for quantitatively evaluating adsorption behavior.[40] The values of KL and $qmax$ can be calculated by plotting $1/Ce$ versus $1/Ce$.[41] Figure 9 shows that the values of correlation coefficients R^2 for the adsorption of all metal ions were more than 0.98. The quantity of metal ions required to create a monolayer on the surface of the substrate ($qmax$ in mg/g) was calculated from (1/intercept), and the constant affinity of the adsorption sites (KL in L/mg) was calculated from 1/(slope $qmax$), as shown in Table 1. The values of RL were calculated for all metal ion concentrations, as shown in Table 2.

Table 1. The values of *qmax* and K*L* for the heavy metal ions with the adsorbent

Heavy metal ion	*qmax*	K*L*
Pb^{2+}	25.30	0.13
Ni^{2+}	16.87	0.10
Co^{2+}	14.45	0.08

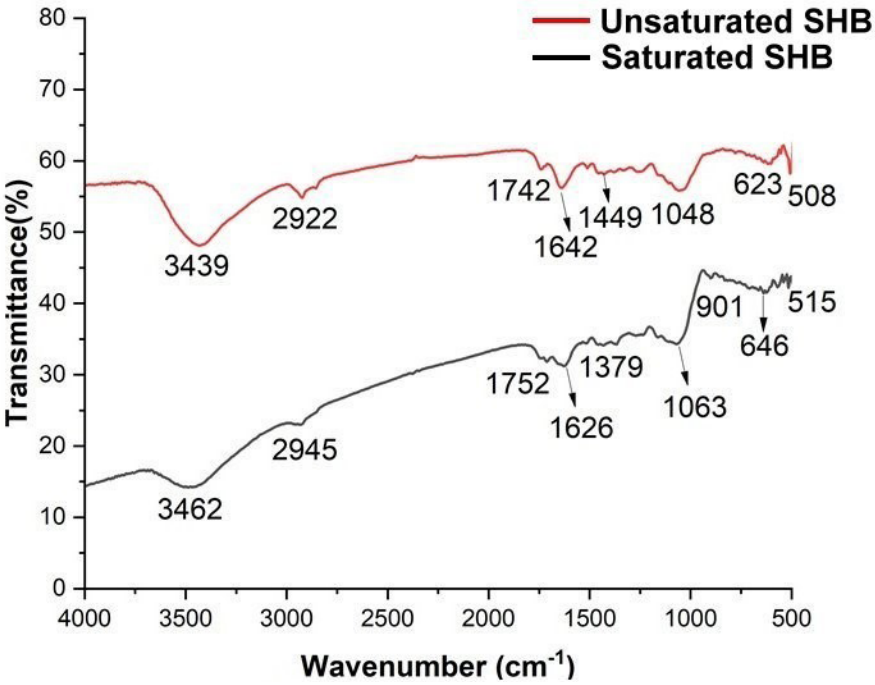

Figure 7. Fourier transform infrared spectroscopy analysis for unsaturated and saturated *Citrullus lanatus* seed husk biomass

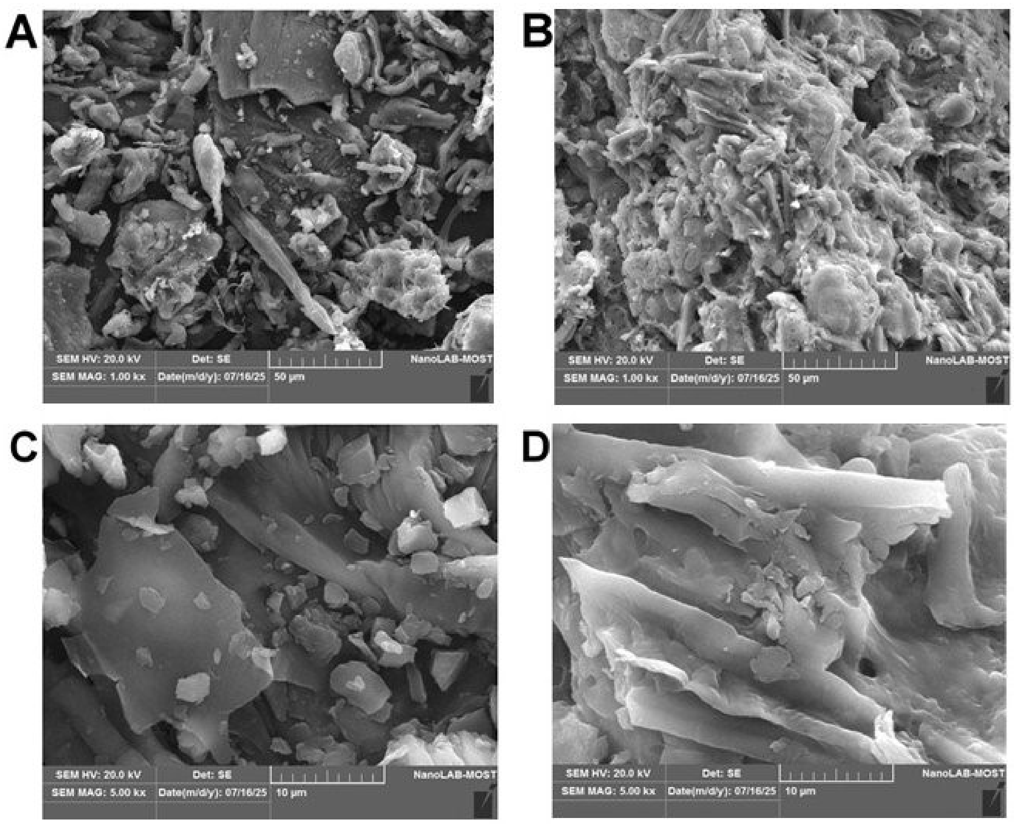

Figure 8. Scanning electron microscopic profile. (A) Native *Citrullus lanatus* seed husk biomass (SHB). Scale bar: 50 µm; magnification: 1,000×. (B) SHB after treatment with metal ions. Scale bar: 50 µm; magnification: 1,000×. (C) Native SHB. Scale bar: 10 µm; magnification: 5,000×. (D) SHB after treatment with metal ions. Scale bar: 10 µm; magnification: 5,000×.

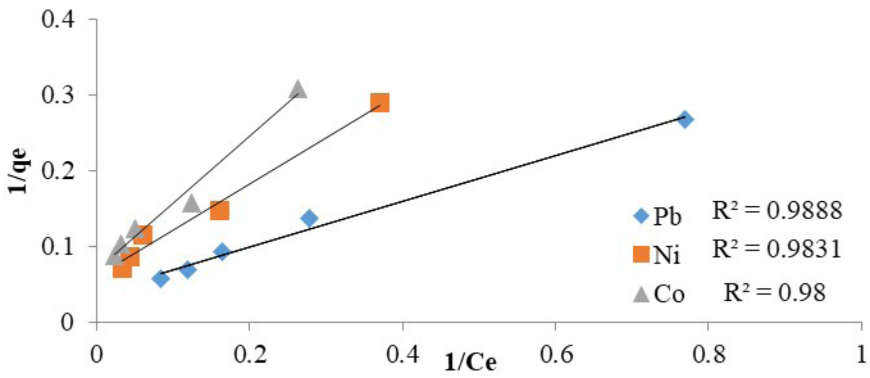

Figure 9. The Langmuir isotherm for eliminating metal ions from aqueous solutions

Table 2. The R*L* values at various concentrations and different heavy metal ions

C*i* (mg/L)	Pb^{2+}	Ni^{2+}	Co^{2+}
20	0.28	0.34	0.39
40	0.16	0.21	0.24
60	0.11	0.15	0.18
80	0.09	0.11	0.14
100	0.07	0.09	0.11

The parameter R*L* denotes the type of isotherm, as defined in Table 3.[18] The interpretation of R*L* values in Table 3 reveals that, for all metal ions and all C*i* concentrations, the type of Langmuir isotherm is favorable as long as 0 < R*L* <1.[42] Therefore, as the R*L* is favorable and the correlation coefficient exceeds 0.95 (Figure 9), all experimental data are fitted to the Langmuir isotherm.

Table 3. The interpretation of R*L* values

R*L* value	Type of isotherm
R*L* > 1	Unfavorable
R*L* = 1	Linear
0 < R*L* < 1	Favorable
R*L* = 0	Irreversible

3.4.2. Freundlich adsorption isotherm

The intercept (log K*f*) and the slope (*n*) from the linear graph log *qe* –log *Ce* are used to obtain the constants K*f* and *n*.[40] It was demonstrated in Figure 10 that ln*qe* vs. ln*Ce* resulted in a high correlation (R^2 > 0.94). The calculated constants are summarized in Table 4, which shows that the order *n* of the total adsorption value indicates that the biosorption mechanisms are in a physical state and exhibit a high degree of heterogeneity.[43,44] Higher *n* values do not necessarily correspond to higher adsorption capacity, which is better reflected by K*f* values. The K*f* values were described by several parameters, i.e., surface specific area, size, and distribution of functional groups on the surfaces. All the heavy metal ion adsorption isotherms studied were found to fit the Freundlich equation, with *n* > 1; patterns of treated biomasses are shown in Figure 10. As shown in Tables 1, 2, and 4, the adsorbent was well fitted to the Langmuir and Freundlich isotherms.

Table 4. The values of *n* and K*f* for the metal ions with the adsorbent

Metal ion	K*f*	*n*
Pb^{2+}	3.04	1.41
Ni^{2+}	2.16	1.84
Co^{2+}	1.95	2.10

3.5. Municipal sewage analysis

This type of water is loaded with large and varied quantities of pollutants from homes, hospitals, food, textile, paint, and tanning factories, as well as other municipal and industrial sources that significantly increase the pollution load in such waters. The results revealed that the effluent water within the RSTP contained heavy metal concentrations (Pb^{2+}, Ni^{2+}, and Co^{2+}) exceeding permissible limits, with values of 2.03, 0.71, and 0.093 mg/L, respectively. The results also indicate the presence of slight differences in the values of these concentrations when examining the water discharged into the Diyala River after treatment. Yet, these concentrations reduced markedly upon SHB treatment (Table 5).

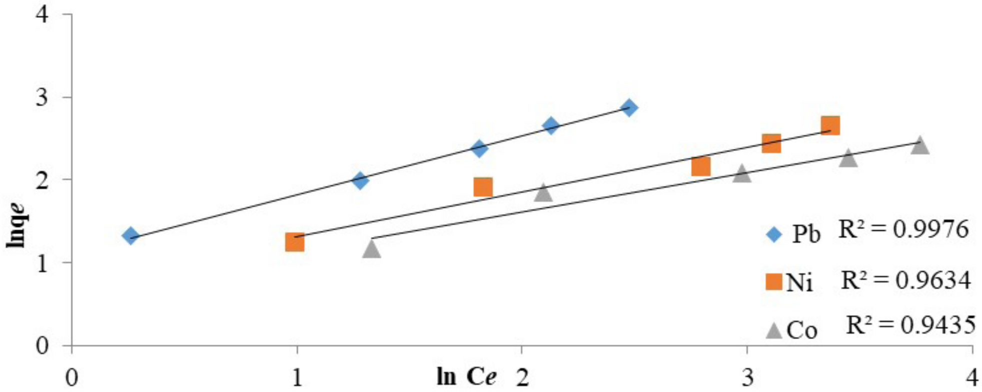

Figure 10. Freundlich isotherm of metal ions removal from aqueous solutions

Table 5. Heavy metal concentrations before and after treatment, and permissible limits for effluent waters (mg/L)

Heavy metal ion	Before plant treatment ± SD	After plant treatment ± SD	After SHB treatment	WHO limits [45]
Pb^{2+}	2.03 ± 0.9	1.74 ± 0.42	Not detected[a]	0.01[b]
Ni^{2+}	0.71 ± 0.08	0.43 ± 0.02	Not detected[a]	0.02[b]
Co^{2+}	0.09 ± 0.01	0.08 ± 0.01	Not detected[a]	0.05[b]

Notes: [a]Detection limits range from about 1 to 100 ppb, depending on the element, according to the manufacturer; [b]mg/L in drinking water.
Abbreviations: SD: Standard deviation; SHB: Seed husk biomass; WHO: World Health Organization.

Discharged water from RSTP contributes to increased concentrations of heavy metals in the Tigris River. This confirms the need for this plant to undergo tertiary treatment to reduce heavy metal concentrations to permissible limits before discharging into the river. At the same time, it emphasizes the urgent need to develop and design a treatment system that uses *C. lanatus* plant residues to treat such hazardous pollutants arriving at this plant. The results are consistent with another study,[46] which demonstrated the deteriorating condition of RSTP in its inability to treat heavy metals.

3.6. Challenges and perspectives

Laboratory studies and field applications exhibit substantial variations in adsorbent biosorption efficiency. Operating conditions, pollutant structure, biosorbent affinity for target metal ions, and other factors affect biosorption efficiency. Wastewater treatment systems handle complex mixtures of organic and inorganic contaminants, spanning a wide range of temperatures, metals, pH levels, and other properties. Seasonality and population affect wastewater amounts. All of these parameters affect biosorption efficiency by affecting *C. lanatus* SHB binding sites and/or potential. Moreover, lab conditions differ from those in the treatment plant. This highlights the need for rigorous scientific studies on the efficacy of biosorbents in real-world sewage and industrial wastewater systems. Research into *C. lanatus* biomass reuse and regeneration across multiple cycles is necessary to sustain this technique. Heavy metals could be recovered while biomass waste is reduced. Date pits, common reed shoots, banana, orange, and pomegranate peels, and other plant leftovers can be reused. These materials can adsorb heavy metal ions over several cycles without losing their heavy metal-binding capacity, thereby enhancing this method. Implementing this plan is crucial for environmental safety and health.

C. lanatus SHB's exceptional metal ion removal ability suggests it could be used as an eco-friendly, cost-effective material to address this type of pollution without harmful consequences. Future scientific research should prioritize the extension and dissemination of these plant remnants in the field. In addition to heavy metals, biosorption technology can reduce pollutants such as dyes and certain organic compounds to an acceptable level. The costs of conventional treatment methods, such as chemicals and pyrolysis, should also be considered.

Sustainable Development Goal 6 and 14 can be reached by using biosorption technology instead of chemical or energy-intensive treatments. Effective adsorption materials like activated carbon are expensive to create. Plant residues are cheaper to produce and prepare while maintaining similar efficacy. Finally, treatment plants can use *C. lanatus* SHB to remove heavy metal ions and mitigate the risks associated with polluted water.

4. Conclusion

The current study evaluates the feasibility of using *C. lanatus* SHB as a biosorbent for heavy metal ion elimination from aqueous solutions and municipal wastewater at the RSTP. *C. lanatus* SHB was selected for its local abundance and low cost, as well as its ability to adsorb metal ions from the surrounding environment. Furthermore, the seed husks of this plant cannot be used as animal feed and are therefore treated as municipal waste or incinerated, both of which increase overall pollution levels. The cellular structure of these husks and their cellulose fibers, rich in active binding groups, make them suitable for adsorbing heavy metal ions from polluted water, thereby reducing their concentration and hazardous effects. The biosorption experiment was conducted under practical conditions with low application costs, including neutral pH, moderate temperature, and a retention time of less than half an hour. The adsorption mechanism and behavior were described using the Langmuir and the Freundlich isotherm models. *C. lanatus* SHB has demonstrated great efficiency, environmental safety, and extremely low cost as a promising low-cost biosorbent for biosorption of heavy metal ions from municipal and industrial wastewater. Analysis of the water samples after RSTP revealed unacceptable heavy metal concentrations, indicating that the treatment process was inefficient for this pollutant.

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Conflict of interest

The author declares no conflicts of interest.

Author contributions

This is a single-authored paper.

Availability of data

The data that support the findings of this study are available from the corresponding author upon reasonable request.

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