

Recovery of Nickel By Synthesized Chitin-Alginate Beads

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Abstract: Nickel contamination of water has arisen as a significant environmental problem because of its toxicity, persistence and bioaccumulation properties. In the present study, the removal and recovery of Ni^{2+} ions from the aqueous solution have been investigated by using eco-friendly and cost-effective bioadsorbent- synthesized chitin-alginate beads. The beads were synthesized by ionotropic gelation method involving the cross linking of a homogeneous solution of chitin and sodium alginate with calcium chloride solution. The batch adsorption experiments were performed at different pH (2-8), temperature (10-60 °C), contact time (10-100 min), adsorbent dose (0.1-1.0 g), and initial concentration of Nickel (20-200 mg/L). Atomic Absorption Spectrophotometry (AAS) was used to determine Residual Nickel concentration. The results showed that the adsorption efficiency strongly varied with pH, with maximum removal of the Nickel at near-neutral pH where there is less competition for the active adsorption sites between H^+ and Ni^{2+} ions. The adsorption was found to be endothermic, being maximum at high temperature after 60–80 minutes. The percentage removal of Nickel ions improved with an increase in the dose of the adsorbent, while the capacity for adsorption increased with increasing initial metal concentration, but removal efficiency decreased due to saturating the active sites. The adsorption equilibrium data were treated by Langmuir, Freundlich, and Temkin isotherm models and the best fit was obtained from the Langmuir isotherm which indicates that the adsorption is monolayer adsorption on a homogeneous surface. The results of kinetic studies show that the pseudo-second-order model gave the highest correlation coefficient ($R^2 = 1$), which indicated the chemisorption as the predominant adsorption mechanism. The results show that the synthesized chitin-alginate beads have a great potential to be an efficient, sustainable and cost effective Ni adsorbent for wastewater treatment.

Keywords: Batch adsorption; Bioadsorbent; Nickel removal; Chitin-alginate beads; Adsorption isotherms; Kinetics; Wastewater treatment

I. INTRODUCTION

In recent years, heavy metal pollution has emerged as a severe environmental problem, largely as a result of industrialization, urbanization and other human activities. Nickel (Ni) is one of the most well-known heavy metals that is extensively used in electroplating, stainless steel manufacturing, battery production, mining and alloying industries. The persistent release of untreated or semi-treated industrial wastewater into aquatic systems has resulted in high levels of Nickel which adversely affect aquatic life and human health. The exposure to Nickel can lead to allergic dermatitis, lung fibrosis, damage to kidney, respiratory diseases, and even carcinogenic effects, making it necessary to effectively remove it from wastewater. Conventional techniques used for heavy metal removal include chemical precipitation, ion exchange, membrane filtration, reverse osmosis and electro-chemical techniques. While these methods are commonly employed, they are not without drawbacks including high cost, secondary pollution, sludge disposal and low efficiency at low metal concentrations. In this regard, adsorption is one of the most attractive and effective techniques due to its



simplicity, low cost, efficiency, and environmental friendliness. Recently, bio-based adsorbents have attracted significant interest due to their renewable, biodegradable and abundant nature. One such bio-based adsorbent is chitin, a biopolymer derived from natural sources such as crustacean shells (shrimp and crab), which exhibits remarkable adsorption capability because of the presence of functional groups such as amino ($-NH_2$) and hydroxyl ($-OH$) that allow binding of metal ions through complexation and ion exchange. However, there are some drawbacks associated with the use of chitin alone, such as its poor mechanical properties.

In order to address these issues, chitin is often combined with sodium alginate, a natural biopolymer derived from brown seaweed. Alginate is a highly gel-forming polymer in the presence of divalent ions like calcium (Ca^{2+}), leading to the formation of porous beads. Chitin's presence in alginate beads improves the mechanical stability, surface area and adsorptive capacity of the composite. The chitin-alginate beads demonstrate enhanced mechanical strength, reusability and separation efficiency from the water, making them ideal candidates for water treatment. Hence, the current research is directed towards the fabrication and utilisation of chitin-alginate beads as a potential bioadsorbent for the recovery of Nickel ions from wastewater. This research examines the effects of various operational factors on the adsorption process and explores the mechanism of adsorption using equilibrium, isotherm and kinetic models. The results will provide insights for the development of effective and ecologically-friendly technologies for the removal of heavy metals from water.

II. MATERIALS AND METHODS

2.1 Chemicals and Reagents

Low molecular weight chitin, sodium alginate and calcium chloride ($CaCl_2$) were purchased commercially and used as received. The other chemicals and reagents used in the study were of analytical grade Nickel sulfate pentahydrate ($NiSO_4 \cdot 5H_2O$), sodium hydroxide ($NaOH$) and hydrochloric acid (HCl). All solutions were made with double distilled water in the experiments. Accurate mass of Nickel sulfate pentahydrate ($NiSO_4 \cdot 5H_2O$) was weighed and dissolved in distilled water to get a stock solution of Nickel ions (1000mg/L). Solutions of desired concentration were obtained using the stock solution to appropriate dilutions.

2.2 Preparation of Chitin–Alginate Beads

Ionotropic gelation method was used for the synthesis of chitin–alginate beads. The ratio of chitin and sodium alginate was determined and placed in distilled water with constant agitation to make a uniform slurry. The prepared solution was then poured into a burette and poured into 0.1 M calcium chloride ($CaCl_2$) solution dropwise. When sodium alginate was contacted with the calcium chloride solution it became ionic cross-linked with calcium ions (Ca^{2+}) to form spherical chitin–alginate beads. Beads were left to gel for 24 hours in the $CaCl_2$ solution in order to obtain complete gelation and structural stability. Then the beads were washed with double distilled water, to get rid of excess calcium ions and unreacted material. The synthesized beads were kept in double distilled water at ambient temperature for future experiments.



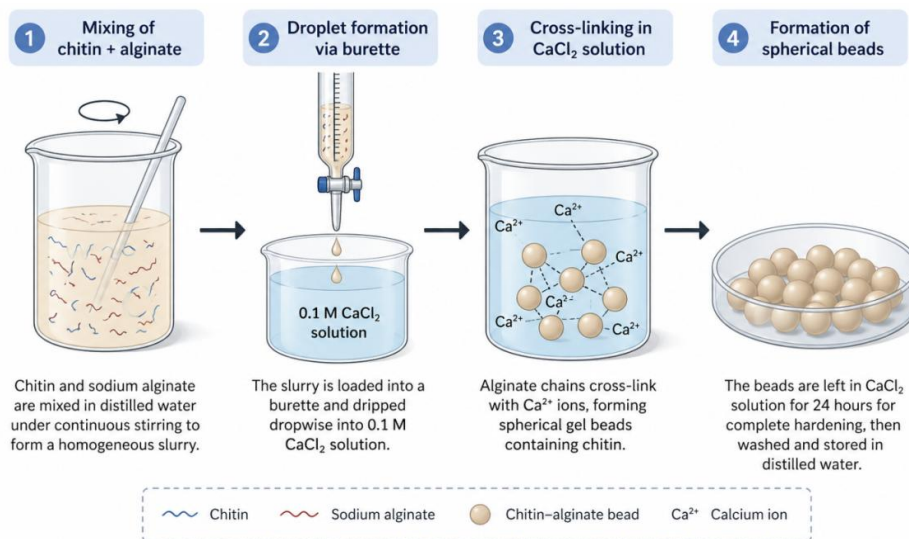


Figure 1: Schematic Representation of Bead Formation

2.3 Preparation of Adsorbate Solution

A 20 mg/L Nickel solution was prepared from the 1000 mg/L stock solution and used as the primary adsorbate for batch adsorption experiments. A series of standard Nickel solutions of various concentrations were also prepared using the stock solution for the preparation of calibration curve for Atomic Absorption Spectrophotometric (AAS) analysis.

2.4 Batch Adsorption Experiments

Batch adsorption experiments were carried out in 150 mL Erlenmeyer flask in a temperature controlled orbital shaker incubator. The synthesized beads of chitin–alginate were washed with double distilled water before every experiment and then gently dried using Whatman filter paper. The amount of chitin–alginate beads used in each experimental run was 0.1 g of beads in 50 mL of Nickel solution, with an initial concentration of 20 mg/L, except in the optimisation studies of the adsorbent dose and initial concentration of Nickel solution. The pH of the solution was adjusted with the help of 0.1 M NaOH and 0.1 M HCl solutions. The flasks were shaken at 100 rpm and kept at constant temperature of 30°C. The predetermined contact time was then over and adsorbent was separated from the solution by filtration by Whatman filter paper. Atomic Absorption Spectrophotometer (AAS) was used to measure the residual concentration of the Nickel ion in the filtrate.

2.5 Data Analysis

The equilibrium adsorption capacity of chitin–alginate beads was obtained from the equation below:

$$q_e = \frac{(C_0 - C_e)V}{m}$$

Where:

- C_0 = initial Nickel ion concentration (mg/L)
- C_e = equilibrium Nickel ion concentration (mg/L)
- V = volume of solution (L)
- m = mass of adsorbent (g)

The calculated equilibrium adsorption capacity (q_e) is the amount of Nickel ions adsorbed per unit mass of adsorbent.



2.6 Adsorption Isotherm Studies

To understand the mechanism of adsorption and surface properties of the adsorbent, the equilibrium adsorption data were analyzed using different adsorption isotherm models such as Langmuir, Freundlich and Temkin isotherms. The Langmuir isotherm is used when the adsorption takes place on a uniform surface and is monolayer adsorption in which the number of adsorption sites is limited and the same. The linear form of the Langmuir equation is given as:

$$\frac{C_e}{q_e} = \frac{1}{q_{\max} K_L} + \frac{C_e}{q_{\max}}$$

The Freundlich isotherm is used for multilayer adsorption on heterogeneous surfaces and can be represented as:

$$\log q_e = \log K_F + \frac{1}{n} \log C_e$$

Temkin isotherm model assumes linear decrease of the heat of adsorption with surface coverage, and also takes into account adsorbent-adsorbate interactions. It is written in linear form as:

$$q_e = RT \ln K_T + \frac{RT}{b_r} \ln C_e$$

The adsorption capacities, adsorption intensities and nature of the adsorption process were evaluated using these isotherm models.

2.7 Adsorption Kinetics Studies

The kinetic studies of adsorption were conducted to check the rate of adsorption and the rate-controlling step of adsorption of Nickel onto chitin-alginate beads. The pseudo-first-order (PFO), pseudo-second-order (PSO) and intra-particle diffusion (IPD) kinetic models were used to analyze the experimental data. In the pseudo-first-order model, the adsorption rate is proportional to the number of unoccupied adsorption sites, and can be represented as follows:

$$\log(q_e - q_t) = \log q_e - \frac{k_1}{2.303} t$$

The pseudo-second order model assumes that chemisorption is the rate limiting step and it can be written as:

$$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{t}{q_e}$$

The intra-particle diffusion (Weber-Morris) model was used to assess the contribution of pore diffusion during adsorption:

$$q_t = k_{id} t^{0.5} + C$$

Correlation coefficient (R²) values were used to assess the suitability of each of the kinetic models. The greater the R² values, the closer the fit between the experimental and theoretical data.

III. RESULTS AND DISCUSSION

The operation condition parameters such as pH, temperature, contact time, adsorbent dose, and initial Nickel ion concentration were adopted to evaluate the adsorption behaviour of Nickel ions onto the synthesized chitin-alginate beads. Further interpretation of adsorption efficiency and adsorption mechanism was done by using adsorption isotherm and kinetic models.

3.1 Effect of pH

The adsorption of Nickel ions to the beads of chitin-alginate is shown in Figure 2 as a function of pH. The adsorption efficiency increased gradually as the pH (acidic) increased to near neutral and maximum adsorption was found at pH 6-7. These were the low pH values where the number of H⁺ ions in the solution was high and the functional groups on the surface of the adsorbent are protonated. Thus, hydrogen ions competed with Nickel ions for available adsorption sites, which resulted in a decrease in the efficiency of adsorption. With the rise in pH, the concentration of H⁺ ions went down and more negatively charged functional groups like -OH and -NH₂ groups were available to bind to the Nickel



ions by electrostatic attraction and complexation mechanism respectively. Beyond neutral pH however, no significant increase in adsorption was seen due to the possible precipitation of Nickel hydroxides under alkaline conditions. This has been recorded in previous studies with alginate based biosorbent for heavy metal removal.

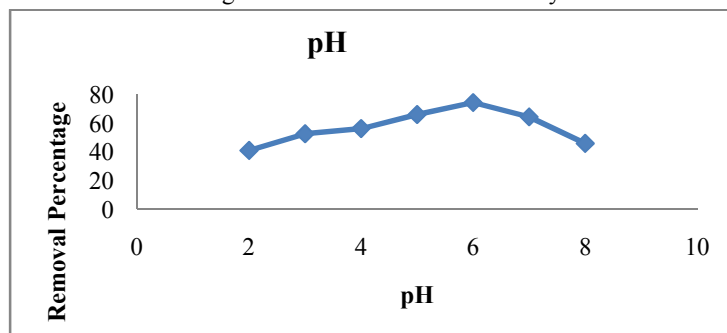


Figure 2: Influence of pH on Nickel ion adsorption by Chitin-Alginate Beads

3.2 Effect of Temperature

The adsorption of Nickel with the influence of temperature is presented in Figure 3. The adsorption capacity was found to be higher with a rise in temperature from 10°C to 60°C, which showed that the adsorption process was endothermic in nature. Higher mobility of Nickel ions in solution at higher temperatures and better interaction between adsorbate and active adsorption sites available in the chitin-alginate beads may contribute to the increase of adsorption at higher temperatures. It is also possible that higher temperatures could lead to more pore diffusion and to the activation of more adsorption sites on the surface of the adsorbent. The improvement in the efficiency of adsorption with temperature may indicate that chemisorption plays a significant role in the adsorption mechanism.

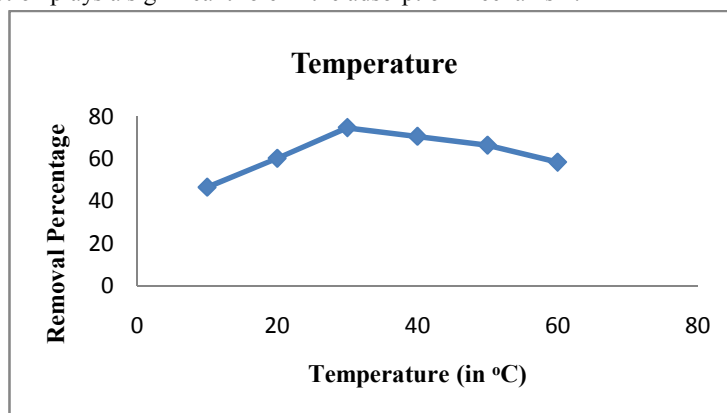


Figure 3: Influence of Temperature on Nickel ion Adsorption by Chitin-Alginate Beads

3.3 Effect of Contact Time

Figure 4 shows how contact time affects the adsorption of Nickel. Initially in the experiment, a large number of adsorption sites were available and hence the adsorption increased rapidly. The adsorption rate was seen to decrease as the contact time progressed and equilibrium was obtained in about 20-30 minutes. However, when the adsorption time is over 50 minutes, no obvious increase in adsorption was seen as most of the active sites were occupied by Nickel ions. The first fast adsorption process might be related to surface adsorption while the second slow adsorption process might be related to the diffusion of the Nickel ions into the internal pores of the chitin-alginate beads.



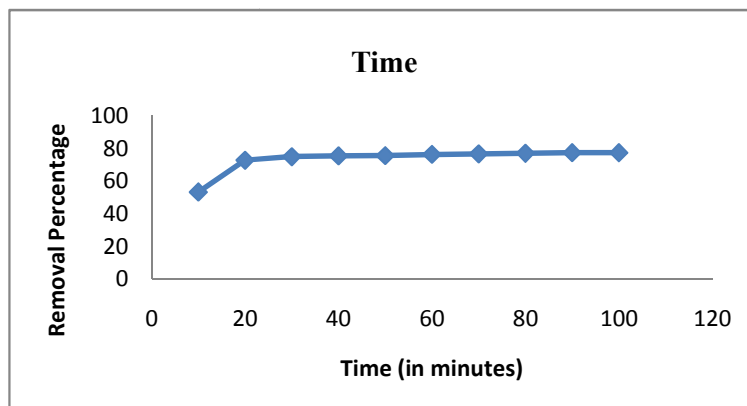


Figure 4: Influence of Contact Time on Nickel ion Adsorption by Chitin-Alginate Beads

3.4 Effect of Adsorbent Dose

The influence of the dose of adsorbent on the removal of Nickel ions was investigated and the results are presented in Figure 5. The percentage removal of Nickel ions was found to be increasing with the increase in the adsorbent dose from 0.1 g to 1.0 g. This rise can be attributed to increase in the number of active adsorption sites and an increase in the surface area at higher doses of the adsorbent. More functional groups were obtained to interact with the Nickel ions, resulting in enhanced adsorption efficiency. As the dosage increases, however, absorption capacity per unit mass of adsorbent dropped because of absorbing site overlapping and aggregating, decreasing the effective surface area available for adsorption.

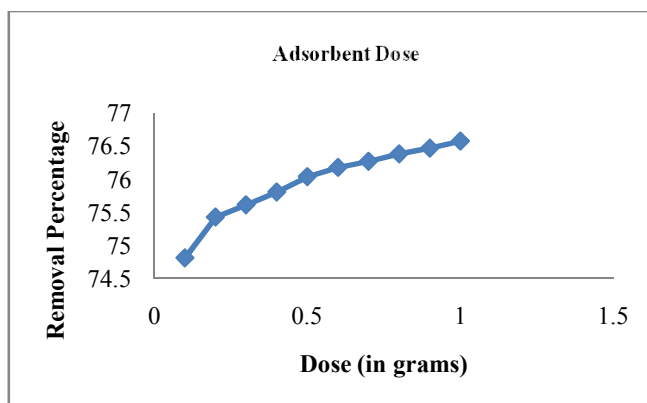


Figure 5: Influence of Adsorbent dose on Nickel Ion Adsorption by Chitin-Alginate Beads

3.5 Effect of Initial Nickel Ion Concentration

The effects of initial Ni(II) concentration were studied. The effect of initial nickel ion concentration was examined. The influence of initial concentration of Nickel ion on the adsorption is shown in Figure 6. This was due to the higher driving force for transferring the mass between the aqueous phase and the adsorbent surface, which led to the increase in adsorption capacity with the increase in initial Nickel concentration. For lower nickel concentration, the number of available active adsorption sites was enough and consequently, the removal efficiency was higher. The percentage removal on the other hand, declined with increasing concentration as the number of adsorption sites was gradually filled up. This behaviour suggests that the chitin-alginate beads adsorption capacity is related to the number of free binding sites on the adsorbent surface.



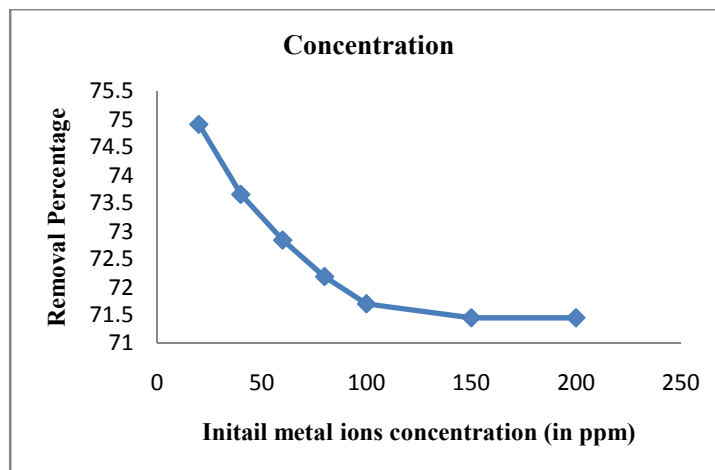


Figure 6: Influence of Initial Nickel Ion Concentration on Adsorption by Chitin-Alginate Beads

3.6 Adsorption Isotherm Studies

The adsorption behavior of Ni^{2+} ions onto chitin-alginate beads was evaluated by using the following adsorption isotherm models: Langmuir adsorption model, Freundlich adsorption model and Temkin adsorption model.

Table 1: Various Isotherm Parameters for Nickel Ions Adsorption onto Chitin-Alginate Beads

Model	R ²	Parameter	Units	Value	Implication
Langmuir	0.736	q_{max} (Maximum Monolayer Capacity)	mg/g	434.78	Indicates high monolayer adsorption capacity
		K_L (Langmuir Constant)	L/mg	0.003	Suggests favorable adsorption affinity
Freundlich	0.999	K_F (Adsorption Capacity)	$(\text{mg/g})(\text{L/mg})^{1/n}$	1.67	Indicates good adsorption capacity on heterogeneous surface
		n (Adsorption Intensity)	—	1.08	$n > 1$ indicates favorable adsorption process
Temkin	0.8833	K_T (Binding Constant)	L/mg	0.186	Reflects adsorbent-adsorbate interactions
		b_T (Heat of Adsorption)	J/mol	25.338	Suggests chemisorption tendency during adsorption

Interpretation

The isotherm model that best correlated closely with the experimental data was Freundlich model ($R^2 = 0.999$), indicating that the adsorption of Ni^{2+} ions onto the chitin-alginate beads was monolayer adsorption on a heterogeneous surface. The Langmuir isotherm plot for Nickel adsorption on chitin-alginate beads shows a moderate linear relationship ($R^2 = 0.736$), indicating that the adsorption process is not purely monolayer and deviates from ideal Langmuir assumptions. The Temkin model showed that the adsorbent-adsorbate interaction and the decrease in the heat of adsorption with surface coverage exist.

3.6.1 Langmuir Isotherm

It is observed that the Langmuir isotherm model is in good agreement with the experimental data, suggesting the presence of a homogeneous surface of the adsorbent for the adsorption of Nickel ions in a monolayer. This model assumes adsorption takes place on particular identical sites with no interaction between the adsorbed ions. The good value of the fitting factor of the Langmuir model indicates that the Langmuir active adsorption sites are uniformly distributed on the surface of synthesized chitin-alginate beads.



3.6.2 Freundlich Isotherm

The Freundlich isotherm model was also well fitted to the adsorption process which suggested heterogeneous surface characteristic and multilayer adsorption behavior. The Freundlich constant showed good adsorption intensity, indicating that the beads of chitin–alginate possess adsorption sites of various adsorption energies.

3.6.3 Temkin Isotherm

The Temkin isotherm model indicated that there was interaction between the surface of the adsorbent and Nickel ions. The model is based on the assumption that the heat of adsorption is linearly decreasing with the increase in surface coverage, which results from the interaction of the adsorbent–adsorbate. The applicability of the Temkin model also suggests the possibility of chemical interactions between the molecules during the adsorption process.

3.7 Adsorption Kinetics

The kinetic study of adsorption was conducted to elucidate the adsorption mechanism and rate controlling step during the adsorption of Ni(II) ions on chitin–alginate beads.

3.7.1 Pseudo-First-Order (PFO) Model

The pseudo-first order kinetic model gave a relatively poor correlation coefficient ($R^2 = 0.7175$) and the kinetic order (rate constant value) was 0.0704 which was not in good agreement with the experimental data. The relatively low R^2 value indicates that any adsorption process is not only a physical adsorption process and cannot completely follow the pseudo-first-order assumption.

Table 2: Pseudo-First-Order (PFO) Kinetic Parameters for Nickel Adsorption

Parameter	Value
R^2	0.7175
Rate Constant (k_1)	0.0704

3.7.2 Pseudo-Second-Order (PSO) Model

The pseudo-second-order kinetic model gave the best fit with the experimental data with the highest value of R^2 (0.999) and the rate constant value of 0.1272. The good agreement of the PSO model suggests that the chemisorption mechanism is the most predominant one affecting the adsorption process. This indicates that the adsorption could be due to electron sharing/exchange between Nickel ions and functional groups present on the surface of chitin–alginate beads.

Table 3: Pseudo-Second-Order (PSO) Kinetic Parameters for Nickel Adsorption

Parameter	Value
R^2	0.999
Rate Constant (k_2)	0.1272

3.7.3 Intra-Particle Diffusion (IPD) Model

The intra-particle diffusion model gave a relatively high value of $R^2 = 0.9213$ with the rate constant value of 0.0766, which suggested that the intra-particle diffusion was responsible for the adsorption process. But because the correlation coefficient was lower than the pseudo-second order model, the intra-particle diffusion was not the only rate controlling step. Overall adsorption mechanism consisted of both surface adsorption and pore diffusion process.

Table 4: Intra-Particle Diffusion (IPD) Kinetic Parameters for Nickel Adsorption

Parameter	Value
R^2	0.9213
Rate Constant (k_{id})	0.0766

IV. CONCLUSION

This study has successfully demonstrated the feasibility of using synthesized chitin–alginate beads as an effective biosorbent for removal and recovery of Nickel ions from water. The adsorption study demonstrated that the process is affected by several operational variables including pH, temperature, contact time, adsorbent dose and initial Nickel



concentration. Notably, pH played an important role in the process and the highest adsorption occurred at near-neutral conditions owing to the presence of maximum active sites and absence of hydrogen ions competing for active sites. Higher temperatures enhanced adsorption efficiency, revealing an endothermic adsorption process, and equilibrium was quickly reached within an optimum contact time of about 60-80 minutes. The adsorbent dose positively affected the percentage removal, but the adsorption capacity (per unit mass) diminished owing to site blocking and aggregation phenomena. Increasing the initial Nickel concentration also led to an increase in adsorption capacity but a decrease in percentage removal, as the sites are saturated.

Isotherm studies indicated that the Langmuir model was the most applicable in describing the adsorption process, which suggested that a monolayer of metal was formed on a homogeneous surface with a finite number of identical sites. The Freundlich and Temkin models also showed the heterogeneity of the surface and the interactions between the adsorbent and the adsorbate. Kinetic analysis indicated that the pseudo-second-order model with an excellent correlation coefficient ($R^2 = 1$) was the best fit, confirming that the adsorption process is controlled by chemisorption. This study also demonstrated intra-particle diffusion, but it was not the only step in the rate-limiting process. In conclusion, the research shows that chitin - alginate beads are an efficient, cheap, environmentally friendly and sustainable adsorbent with high adsorption capacity for Nickel. The simplicity of preparation, high mechanical stability and reusability render them promising for application in water treatment and metal recovery. Further research should consider recycling, scaling up and application in industrial wastewaters to determine their commercial potential.

Declarations

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Authors' Contribution: Equal contribution

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