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Solvothermal Development of Chitosan-Based Adsorbent for the Remediation of Nickel (II) Ions

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A B S T R A C T

Industrial processes that discharge heavy metals into the environment are a growing environmental crisis because of the dangers they pose to aquatic life and human health. Nickel (Ni) is one of the most dangerous metals due to its extreme toxicity and its ability to bioaccumulate in food chains. Severe health complications may develop from chronic exposure to nickel. Chemical precipitation, ion exchange, and reverse osmosis are traditional methods used to remove Ni(II) ions from wastewater. Nonetheless, these approaches have drawbacks, such as elevated operational costs, excessive chemical consumption, and inefficiency in treating low-concentration wastewater. The effectiveness of solvothermally synthesised chitosan as an adsorbent for the removal of Ni(II) ions from aqueous solutions was examined in this work. Acidity, adsorbent concentration, and contact duration were all carefully measured and analyzed. Optimal adsorption occurred at pH 6, yielding a maximum removal effectiveness of 75% after 75 minutes of contact. An adsorption efficiency of 54.69% was achieved with the optimal adsorbent dose of 0.4 g. Kinetic analysis indicated that the adsorption process followed the Pseudo-Second-Order (PSO) model, with a correlation coefficient (R^2) of 0.999, suggesting that chemisorption prevailed via interactions between Ni(II) ions and the $–NH_2$ and –OH functional groups on the chitosan surface. The adsorption capability of the chitosan was improved by the solvothermal modification, which increased its surface area, porosity, and number of active sites.

Contribution to Sustainable Development Goals (SDGs):
SDG 6 – Clean Water and Sanitation (Wastewater Treatment)
SDG 3 – Good Health and Well-being (Toxicity Reduction)
SDG 12 – Responsible Consumption and Production (Eco-friendly Materials)
SDG 14 – Life Below Water (Prevention of Bioaccumulation)

1. INTRODUCTION

Heavy metals released into the environment from industrial activities pose substantial risks to human health and aquatic ecosystems.[1]. Because nickel (Ni) accumulates in food chains and harms living organisms, it is among the most hazardous heavy metals. When exposed to nickel over an extended period, it can lead to skin irritation, inflammation of the lungs, liver, and kidneys, and other serious health problems. [2],[3]. Reverse osmosis, ion exchange, and chemical precipitation are among the most traditional methods employed to treat wastewater and eliminate Ni(II) ions. Nevertheless, the operational expenses, chemical usage, and efficiency of these conventional procedures can be relatively high. [4],[5]. More and more people are looking to adsorption as a possible solution because it is both cost-effective and environmentally friendly. This technique uses an adsorbent substance to remove heavy metal ions from water-based solutions. Even at low metal concentrations, adsorption is effective, and it's cheap and easy to do. [6], [7]. One of the most promising adsorbents is chitosan, a natural biopolymer derived from chitin, which is abundant in the shells of crustaceans such as shrimp and crabs [8]. Chitosan is biodegradable, non-toxic, and contains amino (-NH_2) groups capable of binding Ni(II) ions through ionic and complexation interactions [9]. These properties make it an attractive option for industrial wastewater treatment. Furthermore, producing chitosan from fishery waste aligns with the circular economy principle, as it transforms waste materials into high-value products [10], [11].

Numerous studies have confirmed chitosan's high adsorption capacity for heavy metals and reported that it effectively removes various metal ions, including Ni(II), through ion exchange and complexation with amino groups [12]. demonstrated that structural modifications such as nanoparticle formation or the addition of functional groups can enhance its adsorption capability [13]. Despite its advantages, chitosan's performance can still be improved. One potential enhancement involves solvothermal modification, a process that subjects the polymer to high temperatures and pressures in a solvent. This modification enhances particle uniformity, surface area, and structural stability, thereby improving Ni(II) adsorption capacity.

Studies on solvothermal modification have revealed promising outcomes[14] that chitosan treated by the solvothermal method exhibited a larger surface area and higher Ni(II) adsorption efficiency than unmodified chitosan. found that this modification improved the mechanical strength and stability of chitosan during adsorption [15]. In addition to modification, several parameters affect adsorption efficiency, including solution pH and adsorbent dosage. The pH level influences the speciation of metal ions, which in turn determines their interaction with chitosan [16]. The optimal pH for Ni(II) adsorption onto chitosan lies between 5 and 7, where Ni(II) ions are in a form most reactive toward amino groups.

Additionally, increasing the amount of adsorbent enhances metal uptake by providing more active binding sites. However, beyond a certain threshold, further increases in adsorbent dosage yield minimal improvements due to surface saturation [17]. The research aims to synthesise chitosan via the solvothermal method and to investigate the effects of pH, adsorbent dosage, and contact time on the efficiency of Ni(II) ion adsorption.

2. MATERIALS AND METHODS

2.1. Materials and Equipment

The following substances were utilized in this research: Chitosan (DD 70%), Acetic Acid (CH_3COOH), Sodium Hydroxide (NaOH), Hydrochloric Acid (HCl, 37%), Nickel(II) Sulfate Hexahydrate ($NiSO_4 \cdot 6H_2O$, Pudak), Sulfuric Acid (H_2SO_4), Sodium Tartrate ($Na_2C_4H_4O_6$, technical grade), Dimethylglyoxime (DMG, Merck), Potassium Persulfate ($K_2S_2O_8$, Merck), and Potassium Bromide (KBr, Merck). One tool that was utilized was a UV-Vis Spectrophotometer, namely a Thermo Scientific Genesys 50.

2.2. Preparation of Chitosan via the Solvothermal Method

To dissolve 1.5 g of chitosan, a 1:1 (v/v) solution was prepared using 2% (v/v) acetic acid and 1% (w/v) sodium tripolyphosphate (STPP). The mixture was shaken continuously at 120 rpm and 50°C for 30 minutes to ensure homogeneity. Afterwards, the homogeneous mixture was heated to 180°C under solvothermal conditions for 6 hours in a Teflon-lined stainless steel autoclave. After the procedure was completed, the autoclave was lowered to a normal temperature. Gathering and analyzing the solid product followed.

2.3. Analysis of Ni(II) Using a UV-Vis Spectrophotometer

The quantification of Ni(II) was conducted using a UV-Vis spectrophotometer following the procedure outlined by Kristianto et al. (2019)[18]. In a 50 mL volumetric flask, a 2 mL sample aliquot was added, followed by 1 mL of 1 M H_2SO_4, 2 mL of 20% $Na_2C_4H_4O_6$, 8 mL of 5% $K_2S_2O_8$, 0.5 mL of 1% alkaline DMG solution, and 2.5 mL of 5 N NaOH. Distilled water was added to dissolve the combination to the calibration mark. A UV-Vis spectrophotometer was used to measure absorbance at 470 nm after the samples were allowed to stand for 30 minutes to permit colour development. The interaction between Ni(II) and dimethylglyoxime (DMG) produced a red complex, which was used to determine Ni(II) (Figure 1). Adding NaOH intensified the colour, thereby improving the accuracy of the absorbance measurement.

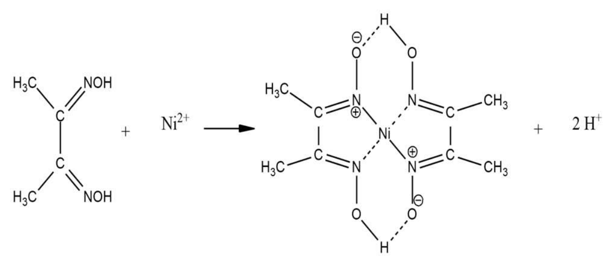

Figure 1. Reaction of Ni(II) Metal with Dimethylglyoxime

2.4. Optimum Conditions for Ni(II) Adsorption

To determine whether solvothermally produced chitosan could effectively remove Ni(II) ions from water, adsorption tests were conducted. Three critical factors were examined to ascertain the ideal settings: solution pH (2, 4, 6, and 8), adsorbent dose (0.2-0.6 g), and contact duration (35, 70, and 150 minutes). The pH was adjusted by adding HNO_t and 0.1 M NaOH. Fifty millilitres of a 25 mg/L Ni(II) solution and 0.1 gram of chitosan adsorbent were used in each experiment. To achieve equilibrium, the mixes were agitated at 250 rpm for 15 minutes. We used a UV-Vis spectrophotometer at 470 nm to measure the amount of residual Ni(II) in the solution after filtering the adsorbent from the solution following the adsorption procedure. The following equations were used to compute the adsorption capacity and efficiency.

$$\text{adsorption capacity } (Qe) = \frac{(Co - Ce)}{m}$$

Where:
Q_e = adsorption capacity (mg/g)
V = volume of solution (L)
C_o = initial concentration (mg/L)
C_e = equilibrium concentration (mg/L)
m = mass of adsorbent (g)

$$\text{adsorption efficiency } (\%) = \frac{Co - Ce}{Co} 100\%$$

Where:
C_o = initial concentration (mg/L)
C_e = equilibrium concentration (mg/L)

2.5. Determination of Adsorption Kinetics

The study of how quickly an adsorbent pulls an adsorbate out of a solution is known as adsorption kinetics. This investigation clarifies whether the process is predominantly chemical or physical and sheds light on the mechanism controlling the adsorption rate. The pseudo-second-order model, which is defined as follows, and the pseudo-first-order model, which is expressed as follows, are the two basic models used to describe the kinetic behavior of adsorption.

$$log\, log\, (q_e - q_t) = log\, log\, q_e - K_1 t$$
$$\frac{t}{q_t} = \frac{1}{K_2\, qe^2} + \frac{1}{q_e}$$

Where:
- qt = Amount of adsorbate adsorbed at time *t* (mg/g)
- qe= Amount of adsorbate adsorbed at equilibrium (mg/g)
- K_1= Rate constant of pseudo-first-order adsorption (min^{-1})
- K_2 = Rate constant of pseudo-second-order adsorption (g/mg·min)

Chemical adsorption, in which the adsorbent and adsorbate share or exchange electrons, and physical adsorption, sometimes called physisorption, are the two main kinetic models that can be used to explain the adsorption process.

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3. RESULT AND DISCUSSION

3.1. Optimization of Ni(II) Ion Adsorption Effect of pH

Because chitosan's surface properties are susceptible to the solution pH, it plays an important role in adsorption. The amine groups ($-NH_2$) on chitosan are protonated in acidic conditions (pH 2-4) due to the abundance of hydrogen ions (H^+). Due to protonation, H^+ and Ni(II) ions compete for active sites on the adsorbent surface, thereby reducing chitosan's adsorption effectiveness for Ni(II) ions [19]. On the other hand, an increase in pH increases the concentration of hydroxide ions (OH^-), which, in turn, improves the electrostatic interactions between chitosan and Ni(II) ions. As a result, the total adsorption capacity is enhanced. [20]

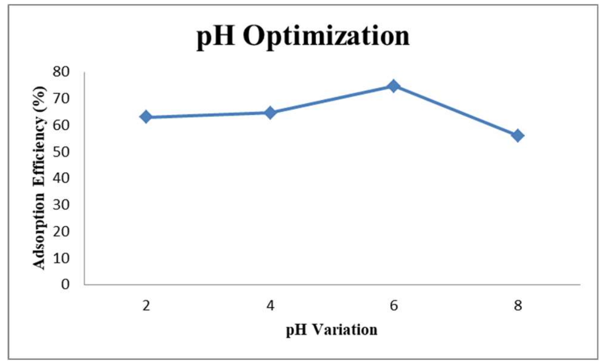

Figure 2. *Optimization of pH Variation for Chitosan*

Nickel hydroxide ($Ni(OH)_2$) starts to precipitate at pH values greater than 6, which makes the adsorption process more difficult. Based on the results, the ideal pH for chitosan to adsorb Ni(II) ions is 6. Hydrogen ions compete with metal ions for active sites at pH values below 6, according to a previous study. On the other hand, stronger electrostatic interactions between Ni(II) ions and the negatively charged chitosan surface occur at higher pH, thereby enhancing adsorption.[21]. To enhance chitosan's adsorption efficacy, its structure is altered during solvothermal synthesis to increase surface area and the number of active sites that can bind metal ions. Improved thermal stability and pore uniformity are two outcomes of applying this method to materials science.[22], [23], ultimately enhancing chitosan's performance in adsorbing Ni(II) ions from aqueous solutions.

3.2. Optimization of Ni(II) Ion Adsorption Effect of Adsorbent Mass

One of the most important factors affecting adsorption is the change in adsorbent mass, which dictates the number of accessible active sites for metal-ion interaction. The adsorption effectiveness of Ni(II) ions (Ni^{2+}) was examined in this work by varying the mass of solvothermally produced chitosan from 0.2 g to 0.6 g. Here is a graph that shows the findings for each adsorbent mass and its accompanying adsorption efficiency:

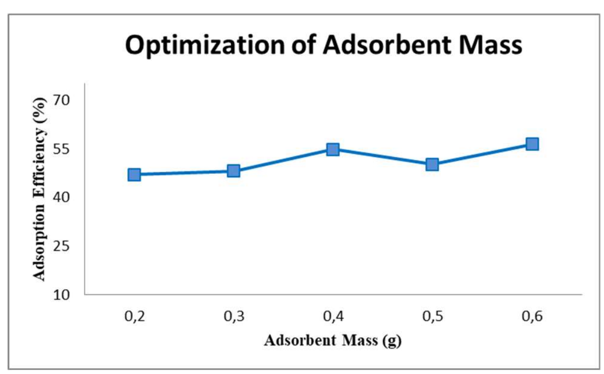

Figure 4. *Optimization of Chitosan Adsorbent Mass on Ni(II) Adsorption Efficiency*

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At 0.2 g of adsorbent mass, the efficiency was 46.95%; at 0.4 g, it rose to 54.69%. It is believed that this improvement is due to more active functional groups (-NH_2 and -OH) on the chitosan surface when the adsorbent mass is greater. These groups are crucial for the complexation of metal ions. [24].

The optimal dose for maximising interaction between Ni(II) ions and the active sites, free of interference from other variables, was 0.4 g of adsorbent, resulting in the maximum adsorption efficiency. Particle agglomeration, which lowers the effective surface area and limits ion diffusion into the adsorbent pores, is likely responsible for the following drop in efficiency to 50.06% at 0.5 g. [25]. Furthermore, saturation of metal ions in the solution may limit interactions with all available active sites [26].

The adsorption efficiency rose to 56.30 per cent at a 0.6 g dose. Still, it did not increase in direct proportion to the amount of adsorbent added, suggesting that many active sites remained unused. This observation aligns with the general adsorption principle that increasing adsorbent mass enhances removal efficiency only up to an optimum point. Beyond that, factors such as agglomeration and system saturation can reduce adsorption performance [27]. Therefore, selecting an appropriate adsorbent mass is vital to achieving optimal adsorption efficiency while maintaining economic feasibility.

3.3. Optimization of Ni(II) Ion Adsorption: Effect of Contact Time and Adsorption Kinetics

The adsorbent's contact time is a critical factor in adsorption effectiveness. The adsorption effectiveness of Ni(II) ions by chitosan-based adsorbents rose from 45 to 75 minutes of contact time, reaching a maximum of 75% at 75 minutes, as seen in Figure 5. The reason for this improvement is that more metal ions are adsorbed onto chitosan, resulting from complexation or ion-exchange interactions between the Ni(II) ions and the functional -NH_2 and -OH groups.

Adsorption efficiency tended to stabilise after 150 minutes, suggesting that the system had reached equilibrium. Since the majority of the active sites had already been taken up by Ni(II) ions, there was no discernible improvement in adsorption performance when the contact period was prolonged.

Consistent with what P. Rahayu and K. Khabibi found, these outcomes (2016)[28], who reported that the optimum contact time for Ni(II) adsorption using a chitosan composite was 60 minutes, after which efficiency remained constant due to equilibrium. Similarly, another study [29] found that after 60 minutes, the adsorption effectiveness of chitosan-activated carbon adsorbents reached a peak of 99%. It may be inferred from this trend that chitosan shows a high initial adsorption rate because to the availability of accessible active sites, but a lower rate when saturation is approached. Accordingly, the optimal conditions for achieving maximal Ni(II) ion adsorption efficiency in this investigation were a contact time of 75 minutes.

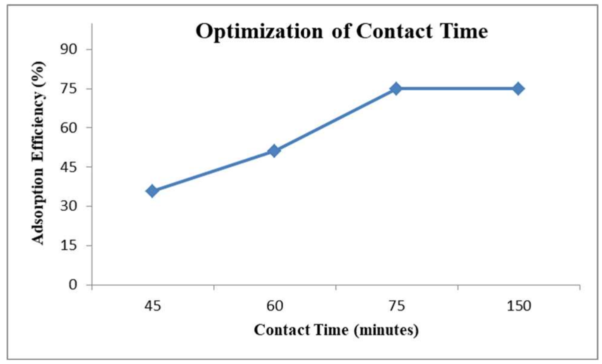

Figure 5. Optimization of Contact Time on the Adsorption Efficiency of Ni(II)

The rate and mechanism of Ni(II) ion adsorption onto the solvothermal chitosan adsorbent were investigated using kinetic analysis. The physical adsorption process was represented by the Pseudo-Second-Order (PSO) model, while the Pseudo-First-Order (PFO) model represented the chemical adsorption process. Both the PFO and PSO models account for chemical interactions between metal ions and the adsorbent's functional groups; however, the PFO model assumes that the adsorption rate is proportional to the number of available active sites on the adsorbent's surface. The equilibrium adsorption capacity (qe), correlation coefficient (R^2), and rate constants (K_1 and K_2) were the kinetic parameters examined in this study.[30]. The kinetic analysis results are summarized in Table 2

Table 2. Kinetic Parameters for Ni(II) Ion Adsorption on Solvothermal Chitosan

Kinetic Model	Parameter	Solvothermal Chitosan
Pseudo-First-Order (PFO)	Rate constant, K_1 (min^{-1})	0.035
	Calculated qe (mg/g)	1.72
	Experimental qe (mg/g)	1.87
	R^2	**0.993**
Pseudo-Second-Order (PSO)	Rate constant, K_2 (g/mg·min)	0.012
	Calculated qe (mg/g)	1.90
	Experimental qe (mg/g)	1.87
	R^2	**0.999**

While both kinetic models accurately describe the adsorption behaviour of Ni(II) ions, the Pseudo-Second-Order (PSO) model provides a better fit to the data. The experimental result of 1.87 mg/g is quite similar to the estimated equilibrium adsorption capacity (qe) of 1.90 mg/g from the PSO model, indicating significant agreement between the two sets of data. The PSO model more faithfully captures the adsorption kinetics, as evidenced by its higher R^2 of 0.999, compared with the Pseudo-First-Order (PFO) model's R^2 of 0.993. This dissimilarity suggests that chemical rather than physical interactions are the primary determinants of adsorption.

Chemical linkages between Ni(II) ions and the active $-NH_2$ and -OH groups on the surface of the chitosan are part of the chemisorption process, which is further supported by the close agreement between the experimental and estimated qe values. The adsorbent is firmly bound to Ni(II) ions as a consequence of these interactions, which most likely take place via electron exchange or coordination bonding. During the first phase of adsorption, Ni(II) adsorption occurs rapidly, as indicated by the rate constant (K_2) of 0.012 g/mg·min. The effective contact between Ni(II) ions and adsorption sites is promoted by the increased surface area and porosity of solvothermally synthesised chitosan, as evidenced by a larger K_2 value [29], indicating that the metal ions interact with the adsorbent's active sites more efficiently. This confirms that solvothermal treatment enhances the surface reactivity of chitosan toward heavy metal ions, as earlier results showed that modified chitosan had higher adsorption rate constants than unmodified chitosan [30].

Consistent with other research, such as [31], our findings show that the PSO model was followed by adsorption of Ni(II) ions onto magnetite-cellulose-chitosan composites, with an R^2 value greater than 0.98. Results showing that the adsorption process was primarily governed by chemical interactions between functional groups and metal ions were also obtained using chitosan-activated carbon adsorbents. The importance of amino and hydroxyl groups in creating strong coordination interactions with metal ions is highlighted by these consistent findings, which together confirm that Ni(II) adsorption on chitosan-based materials often happens via chemisorption.

As a result, solvothermally synthesised chitosan is a reliable adsorbent for Ni(II) ions due to its strong adsorption capacity. When it comes to explaining the adsorption behavior of Ni(II) ions on solvothermal chitosan, the Pseudo-Second-Order (PSO) model is the most appropriate, according to the kinetic data. Chemisorption must be the governing mechanism in this process, as the R^2 value is getting close to 1 and the estimated and observed qe values are in excellent agreement. This material is highly effective at removing heavy metals with short contact times, as evidenced by its high adsorption rate. Thus, solvothermally synthesised chitosan has great promise for use in Ni(II)-containing wastewater treatment systems, and the PSO model provides a solid theoretical foundation for future improvements in biopolymer-based adsorption reactor designs.

4. CONCLUSION

The effectiveness of solvothermal chitosan as an adsorbent for Ni(II) removal from water-based solutions was shown to be pH- and contact time-dependent. To achieve optimal electrostatic attraction and complexation, the ideal pH for the reaction was 6. The optimal adsorbent dose was 0.4 g, yielding 54.69% effectiveness, whereas higher doses led to particle agglomeration and reduced adsorption. According to the Pseudo-Second-Order (PSO) kinetic model, adsorption reached equilibrium at 75 minutes, with an efficiency of 75%. The results show that chemisorption is the dominating process, with an R2 value of 0.999. Chitosan showed great promise as an environmentally benign adsorbent for treating Ni(II)-contaminated wastewater after undergoing solvothermal alteration, which increased its surface area, porosity, and reactivity.

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