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Dependence of adsorption capacity of succinate functionalized linseed for Ni (II) adsorption on different factors

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Abstract: An effective sorbent for removing Ni(II) from aqueous solutions was developed by chemical modification of a polysaccharide from linseed that provided multiple new functional groups and produced a succinate derivative (SRG), which was then converted into sodium salt (Na-SRG). To having good capacity for holding water, it was tested for its ability to remove Ni (II) from solution. The effects of initial concentration of Ni(II), the sorbent dose, temperature, time to contact, and the pH of the metal solution were investigated through batch adsorption tests using Na-SRG. All the tested parameters had prominent effect on the sorption capacity of Na-SRG in removing Ni(II). The Ni(II) reached equilibrium quickly in 30 min. Hence, Na-SRG is sorbent material of great choice to remove metals from polluted water.

Keywords: Adsorption, Linseed, hydrogel, wastewater, pH

1. Introduction

Heavy metals are a major class of environmental pollutants due to their persistence, bioaccumulation, and toxicity. Their longevity in ecosystems is a substantial threat to the ecological balance of the planet and poses a risk to human beings and other organisms that consume them [1]. Of the numerous heavy metals, Ni(II) is a common source of concern as heavy metal contaminants. The use of this metal is prevalent in various industries. Ni(II) is commonly used in the production of nickel-cadmium batteries, as pigments, and in the production of plastics (i.e., toys and jewelery) [2-4].

Ni(II) is valuable in many industrial applications. It is dangerous environmental toxin that exist in various forms, enhancing their bioavailability and associated risks. In response to this risk, agencies such as the WHO, and the United States EPA have promptly regulated permissible levels for these metals in drinking water. Anthropogenic activity in the form of industrial manufacturing, industrial discharges, and waste directly relates to co-exposures and increased environmental concentration that exceed Ni(II) permissible value. Prolonged exposure to this higher level causes serious diseases of critical organs. The development of reliable remediation methods for toxic heavy metals from contaminated waters is of high importance to public health outcomes [5-7].

Union of different types of physicochemical methods has been used to remove heavy metals from contaminated water. These include methods like chemical precipitation [8], reverse osmosis [9], ultrafiltration membrane systems [10], and electrochemical processes [11]. However, due to its many advantages, the method receiving the greatest interest is adsorption [12]. The high enrichment capability of adsorbents combined with simple separation techniques and the ability to use inexpensive eco-friendly materials as sorbents makes it a preferred method compared to others [13]. Typical biosorbents often consist of biological materials, Agro-waste, and activated carbon. One common limitation of many traditional materials is their relatively low sorption capacity, which is driving the impetus for research to develop new, low-cost and environmentally friendly sorbents for water treatment.

Hydrogels based on polysaccharides have attracted a lot of interest in the past years due to their biomedical and industrial applications. These materials can be chemically functionalized using carboxylic acid anhydrides (e.g., succinic anhydride) to enhance their metal-binding ability, making them very exciting sorbents for the sequestration of Ni(II) ions from groundwater leachates and wastewater. Mucilage and polysaccharide-rich materials will also have intrinsic stimuli-responsive, super-porous, superabsorbent, and nontoxic properties, which have led to successful work investigating them for developing effective sustained-release drug delivery systems [14-29]. An example of this is Linseeds (*Linum usitatissimum* L.) that produce a hydrogel (Linseed hydrogel - LSH) which is largely composed of rhamnogalacturonans (RG) and have been effectively used as a sustained-release agent for the encapsulation of the pH responsive drug diclofenac sodium and gastroprotective matrix tablets for antibiotics (moxifloxacin) [30, 31].

The objective of this research is to develop a sustainable, chemically enhanced super-sorbent derived from rhamnogalacturonans (RG) for the efficient removal of Ni(II) ions from aqueous systems. The study focuses on improving the metal-binding performance of RG by converting it into its sodium succinate form (Na-SRG), thereby increasing the availability of active functional groups. The influence of key operating variables, such as initial metal concentration, sorbent dosage, contact time, pH, and temperature will be assessed to determine their roles in enhancing sorption efficiency.

2. Materials and methods

2.1. Materials

RG hydrogel was obtained by extracting it from flaxseed. Flaxseed was collected from a local store - it was manually cleaned by removing debris (dust and trash) and sorting the seeds before conducting any further testing. Prior to extraction, the flaxseed was cleaned and deionized water was used for all experimental steps. All reagents and solvents are of analytical quality or higher and did not require further purification. All glassware was pre-rinsed thoroughly with nitric acid before conducting any experiments.

2.2. Isolation of hydrogel from Linseed

RG extraction from flaxseed began as per previous methods [30]. The flaxseed soaked in deionised water (DI) for 48 h at room temperature and heated to 80°C to allow mucilage to flow out of the seeds. The mucilage was separated from flaxseed using a nylon screen and defatted using *n*-hexane three times. The resulting dissolved mucilage was centrifuged at 4000 rpm for 2 h yielding a solid pellet known as linseed hydrogel (RG). The dried RG was ground finely and screened through a No. 60 screen and stored in airtight containers in desiccators until ready for future use.

2.3. Synthesis of SRG and sorbent Na-SRG

The SRG and Na-SRG was synthesized according to already reported procedure [26].

2.4. Sorption studies

The ideal conditions for the adsorption of Ni(II) ions onto the Na-SRG sorbent and the effect of several important key parameters have been determined using batch adsorption studies.

The five key parameters that were studied were the initial metal concentration, the amount of sorbent used, the solution pH, the contact time, and the temperature. Stock solutions of Ni(II) was prepared by dissolving specific amount of $NiCl_2$. $2H_2O$ into 1 L of DI to produce metal stock solutions of 1000 mg/L. Working solutions at various concentrations were obtained from the stock solution by dilution, and the pH of each final working solution was adjusted to the desired pH using 0.1 M NaOH or HCl solutions. For every experiment, an Erlenmeyer flask filled with 100 mL of a metal ion solution (usually 100 mg/L) was placed on a shaking thermostat and shaken for 30 min at a predetermined temperature. After shaking, the solid sorbent was collected through a filter to isolate any Ni(II) ions left behind in the solution. The concentration of both ions still present in the filtrate was determined using Flame Atomic Absorption Spectroscopy (FAA) and the amount of Ni(II) ions were removed using the sorbent material was calculated according to the standard formulas listed in Equations 1 and 2, where q_e equals the amount of metal ions adsorbed by each sorbent.

$$q_e = \frac{C_i - C_e}{m} \times V \quad (1)$$

$$Percentage\ uptake = \frac{C_i - C_e}{C_i} \times 100 \quad (2)$$

In these equations, the equilibrium sorption capacity is denoted as q_e (mg g^{-1}). In these equations, C_i, and C_e, correspond to the initial and final metal concentrations, respectively (mg L^{-1}); V is the volume of the solution (L), and m, the mass of the adsorbent (g).

2.4.1. Effect of initial metal ion concentration

The effect of the initial metal ion concentration on the sorption performance of Na-SRG was tested. The sorbent (30 mg) was shaken with 100 mL of Ni(II) solution at varying concentrations (20 to 160 mg L^{-1}) for 30 min at 200 rpm and 298 K. The outcomes of these tests were then used to define sorption equilibrium. The outcome of these tests was used to find the optimum initial metal concentration for future experiments.

2.4.2. Effect of sorbent dose

To find the ideal dose of sorbents, different masses of Na-SRG, between 10 and 90 mg, were added to 100 mL solutions of 100 mg L^{-1} of Ni(II) obtained from the previous experiment. The goal of this experiment was to determine the lowest number of sorbents needed to achieve maximum adsorption of metal ions from the aqueous solution.

2.4.3. Effect of pH

The effect of pH on the adsorption efficiency was assessed using 100 mL solutions of Ni(II) (100 mg L^{-1}) using 30 mg of sorbent. These solutions had an initial pH adjusted from 2 to 10 with 1 M HNO_3 or NaOH to determine the best conditions under which to adsorb metals.

2.4.4. Effect of contact time

To study the sorption kinetics and understand the mechanism, experiments were performed by stirring an optimum dose of sorbent (30 mg) in 100 mL of metal solutions at a concentration of 100 mg L^{-1} and pH 6. The contact time was investigated between 5 - 120 min at 298 K. The remaining concentrations of Ni(II) in the supernatant were measured by FAAS, and the data was evaluated using pseudo-first order and pseudo-second order kinetic models.

2.4.5. Effect of temperature

To assess the thermodynamic nature of the sorption process, we examined its temperature dependence. Following the normal procedure, we conducted our tests over a temperature range of 298 to 348 K to explore whether the metal uptake was exothermic or endothermic, and if it was spontaneous or non-spontaneous.

3. Results and discussions

3.1. Effect of initial metal ion concentration

As shown in Figure 1, the amount of metal (mg metal per gram of sorbent, or q_e) that Na-SRG could hold increased with increasing metal ionic concentrations until the amount of available metal ions reached saturation. The Ni(II) was most effectively sorbed at the initial condition of 100 mg L^{-1}, and increasing concentrations of either ion provided no further sorption capacity to Na-SRG. This implies there should be additional work at higher levels of initial metal ionic concentrations to evaluate the sorption performance of Na-SRG. Initially, the increase in sorption capacity was attributed to the increase in available metal ions present and creating a greater concentration gradient that would drive the transfer of metal ions to Na-SRG. The plateau for initial concentrations above 100 mg L^{-1} indicates that the anion-exchangeable sites on the surface of Na-SRG were occupied by Ni(II) ions, thereby limiting additional sorption of cations [14, 32].

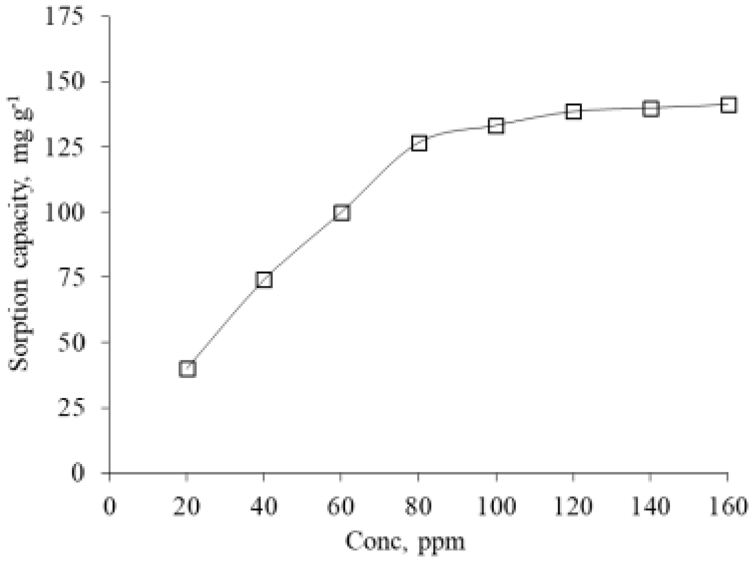

Figure 1: Effect of Ni(II) ions concentration.

3.2. Effect of sorbent dosage

Experimental work was performed to identify the optimal quantity of sorbent necessary to obtain the maximum removal of metals. The findings indicated that the adsorption of Ni(II) with increasing dosages or masses of Na-SRG was enhanced due to the increasing number of available binding sites from the greater quantity of sorbent introduced into the system, but once a correct mass of 30 mg was reached, there was a minimal decline in the rate of metal uptake. This decline is a function of the overall concentration of the metal ions present in the solution; any additional mass of sorbent beyond the threshold of 30 mg had unoccupied binding sites because of an inadequate number of metal ions in the solution [25, 33]. It was determined based on this information that the optimal mass of sorbent to be used for subsequent experimentation will be 30 mg of Na-SRG, as illustrated in Figure 2.

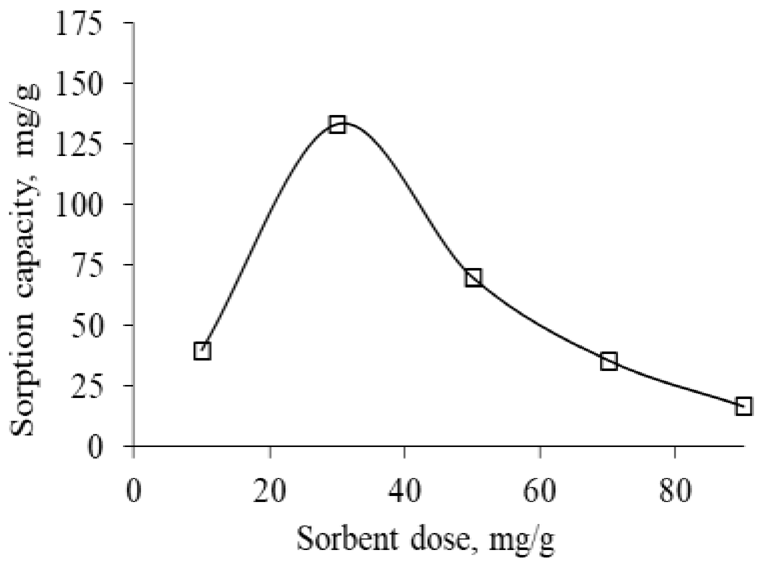

Figure 2: Effect of sorbent dose.

3.3. Effect of pH

Sorption ability for Na-SRG is immensely affected by pH of solution as it controls surface charge and ionization state of functional groups. Ni(II) sorption trials were performed on Na-SRG materials with a pH between 2.0-10.0 (Figure 3). Low adsorption values recorded in acidic mediums are believed to be caused by protonation and conversion of Na-SRG into its acid form (SRG). Vault-like cation exchange sites become blocked from uptake of metals during this formed condition. As pH increased so did Na-SRG sorption ability with maximum sorption accomplished at pH 6.0. Deprotonation of various functional groups and establishing a negative charge when solution pH surpasses the sorbent's point of zero charge (pH_{ZPC}) assists in the rapid increase of metal sorption at this elevated range. The area surrounding the pH_{ZPC} portrays a positive charge meaning there exists limited ability to bind with metal ions; beyond this zone the precipitation of $Ni(OH)_2$ reduces the overall metal ion concentrations available in solution thereby decreasing efficiency for exchange reactions [29, 34]. Thus, pH 6.0 was determined as the optimal condition to perform all remaining sorption tests.

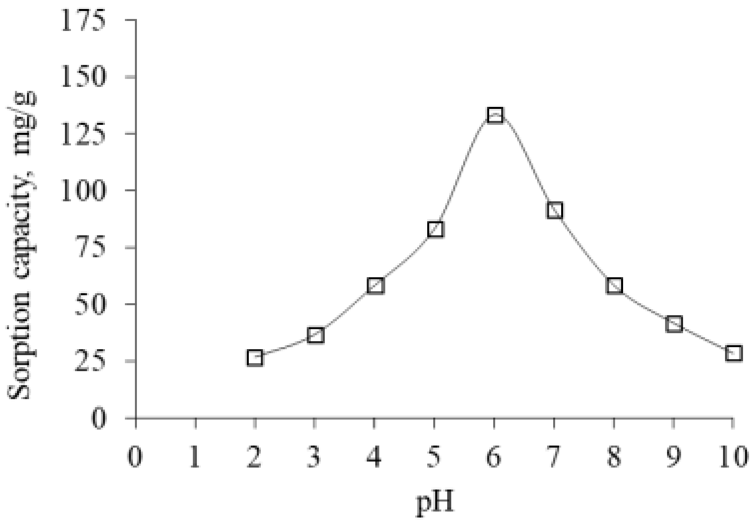

Figure 3: Effect of pH.

3.4. Effect of contact time

The contact duration between the sorbent's active sites and the metal ions in solution has a significant effect on total sorption capacity. Contact time evaluation provides insight into the adsorption mechanism and helps to determine the kinetic model best suited to the material. Contact time across Ni(II) ions were determined for Na-SRG. The contact period that resulted in approximately 85% removal of metal ions (Figure 4), was 30 min, which was the point at which most of the metals where adsorbed. This initial rapid metal uptake occurred due to an abundance of sodium-succinate functional groups on Na-SRG, and the rapid kinetics associated with the ion-exchange process where Na^+ ions are exchanged with Ni(II) ions.

Beyond 30 min, the rate of metal adsorption was significantly reduced as the repulsion between the metal ions already adsorbed and the incoming cations decreased access to unoccupied active sites for further sorption. After 30 min, no significant increases in metal ions adsorbed were observed, which indicates that increasing the contact time will not increase the sorption capacity of Na-SRG [35]. This shows that Na-SRG will achieve an equilibrium rate quickly, making it a practical and beneficial material.

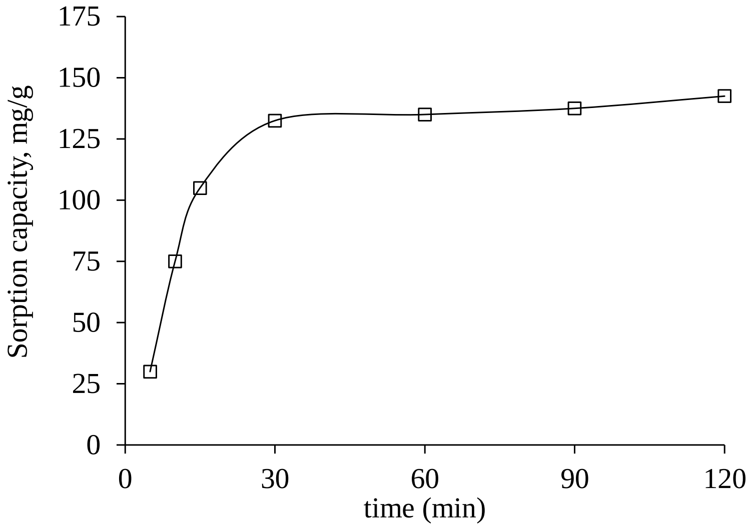

Figure 4: Effect of time.

3.5. Effect of temperature

In this study, the effect of temperature on the performance of Na-SRG for sorption of toxic metals has been investigated to gain insight into the thermodynamics of the sorption process, specifically whether the sorption process is exothermic or endothermic. The experiments were conducted at different temperatures from 298 K to 343 K. As shown in Figure 5, the sorption capacity of the adsorbent was highest at the lowest temperature and decreased as temperatures increased, indicating that the sorption process is exothermic. The increased temperature at which Na^+ ions in the sorbent moved rapidly would restrict the mobility of Ni(II) ions from being able to occupy all the active sites on the surface of the sorbent when the temperatures are lower than 298 K. As the temperature increases, the increased activity of Na^+ ions would reduce the ability of Na^+ ions to undergo effective ion exchange and therefore reduce the amount of metal cations that can be taken up into the sorbent. Based on these data, it can be concluded that the optimal temperature for uptake of Ni(II) ions into the sorbent is 298 K.

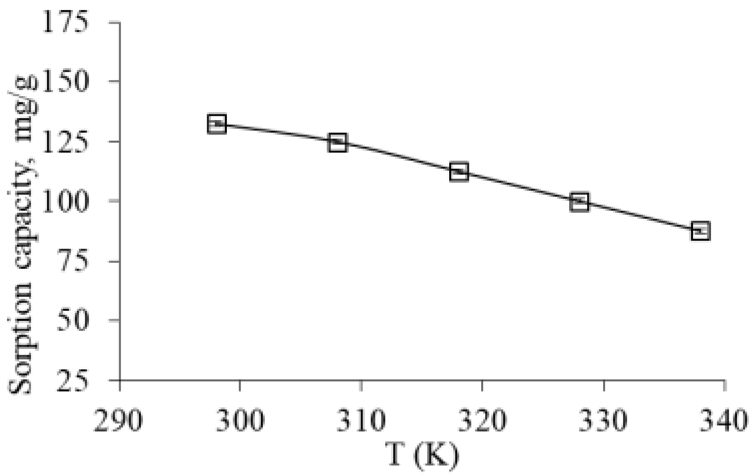

Figure 5: Effect of temperature.

4. Conclusions

This research successfully created a new biosorbent, the sodium salt of succinylated rhamnogalacturonans (Na-SRG), through an esterification procedure. The performance of Na-SRG in taking-up Ni(II) ions from aqueous solutions was significantly influenced by various operational parameters. The kinetics of sorption were rapid, with greater than 85% uptake for both metals occurring in the first 30 min of the process, described most closely by a pseudo-second-order kinetic model implying that chemisorption characterized by ion-exchange dominates the uptake. The measured high sorption capacities and favourable kinetics confirm that Na-SRG is a highly efficient, reusable and super-sorbent with great promising potential for the selective uptake of Ni(II) from contaminated water.

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