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OPTIMIZATION OF ADSORPTION CONDITIONS FOR NI(II)-REMEDIATION FROM POLLUTED WATER USING A SUCCINATE-FUNCTIONALIZED ADSORBENT

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Abstract

Hydrogels are the hydrophilic biomaterials with three-dimensional structure. Their chemical structures can be modified owing to the presence of chemically modifiable functional groups such as carboxylic (-COOH) and hydroxyl (-OH). Consequently in the present study, an acetylated hydrogel from the seeds of Salvia spinosa (SH) was extracted, succinylated and saponified to synthesize an eco-friendly polysaccharide based ion-exchanger (Na-SH) for developing a cost effective sorbent medium towards Ni(II) retrieval from water polluted. The influence of five parameters namely sorbent dose (10-90 mg/100 mL), initial Ni(II) ion concentration (10-250 mg L^{-1}), duration of contact time (5-150 min), temperature (298-338 K) and pH (1-7) on the adsorption capacity was investigated. Considerable amount of Ni(II) was removed using Na-SH adsorbent. The maximum Ni(II) uptake was found at sorbent dosage = 20 mg/100 mL, initial concentration Ni(II) ions= 100 mgL^{-1}, contact time = 30 min, temperature = 298 K, and pH value of 6. Thus, these conditions were selected as best one for the remediation of Ni(II) from polluted water.

INTRODUCTION

Due to numerous human activities and unchecked industrialization, heavy metals are constantly seeping into water supplies. As a result, heavy metal ions pose long-term risks of water pollution in addition to harming global stability [1, 2]. Nickel (Ni(II)) is one of the most dangerous and prevalent heavy metals pollutants for fresh water [3]. Because Ni-Cd batteries and pigment are frequently used, it (Ni(II)) is therefore, widely found in households and industrial waste [4]. Additionally, the World Health Organization, recommended limits of Ni(II) in drinking water is exceeding by mining activities, burning fossil fuels, leather tanneries, metallurgical operations, etc. Humans exposure to Ni(II)-based contaminated water results in the various life threatening diseases [5]. Water purification is therefore regarded as a different strategy to address the global scarcity of drinking water.

Various conventional physical and chemical techniques have been reported for the removal of Ni(II) from both surface and ground water in the past two decades. Due to its economic benefits, selectivity toward speed, efficiency and regenerability, easy handling as well as wide range of adsorbent materials; adsorption followed by ion exchange appeared to be most promising technique [6-10]. Due to their high water absorbancy and non-toxic nature of the polysaccharide based hydrogels, they have extensively been utilized as drug delivery carriers and sorbent for water purification [11-26]. The *Salvia spinosa* hydrogel (SH) has been esterified with acetic anhydride [14] and citric acid [17], the latter also showing some thermal stability and being referred to as a promising material for controlled release of drugs. Therefore, SH can also be esterified with succinic anhydride and then saponified as well. This product (Na-SH) is anticipated to be a sorbent for the ion-exchange of its own ions with heavy metal ones in polluted water.

This study reports the synthesis, purification and characterization of Na-SH as sorbent for Ni(II) ions from a contaminated water. The purpose of present work is to investigate and compare the influences of pH, sorbent dose, initial Ni(II) concentration, contact time and temperature on the uptake capacity of Na-SH. The objective is towards maximizing adsorption condition for the extension of this material to strip off toxic heavy metal ions and dyes in personal care and water environments, to save live's.

2. Materials and methods

2.1. Materials

The seeds of *S. spinosa* were obtained from the local market which was collected from the District Sargodha, Pakistan. All chemicals and reagent employed in this study were of analytical grade (> 99% pure) and used as received. All the reagents and chemicals used in this work were purchased from Riedel-de Haën, Germany and Sigma-Aldrich, USA.

2.2. *S. spinosa* Seed Hydrogel Isolation

Following post-sieving and screening for elimination of undesirable materials, the seeds of *S. spinosa* were soaked in DW at 50 °C for 2.5 h with a muslin cloth and spatula to separate the excreted seed's mucilage (SH). The as-obtained SH was first washed by DW to remove polar impurities, and then washed with n-hexane for triple times to eliminate non-polar substances. SH was extracted after which it served as a starting material to obtain SH via centrifugation (4000 rpm) followed by drying in vacuum oven at 60 °C, screen grinded to pass through no. 60 mesh and stored in air-tight container under vacuum desiccator [19].

2.3. Synthesis of Adsorbent

The adsorbent, i.e., sodium salt of SH was prepared using the procedure established by [27].

2.4. Studies on Sorption

To control the optimum experimental conditions for Na-SH to adsorb as much Ni(II) as possible from polluted water, a serial of batch sorption measurements were carried out at room temperature. Sorption by using Na-SH was found to rely on different operating parameters, such as pH (1-7), sorbent dosage (10-90 mg/100 mL), initial concentration of Ni(II) ions (10-210 mg L^{-1}), contact time (5-150 min), and temperature (298-338 K).

To facilitate the complexation process, Ni(II) salt ($NiCl_2 \cdot 2H_2O$) was first dissolved in deionized water using a 1-L volumetric Erlenmeyer flask to prepare a 1000 ppm (1000 mg L^{-1}) stock solution. The solution was stirred thoroughly to ensure complete dissolution. From this stock, working solutions were prepared by appropriate dilution, and their pH values were adjusted using 0.1 M HCl or 0.5 M NaOH, with continuous monitoring using a calibrated pH meter. Subsequently, 250-mL Erlenmeyer flasks were filled with 100 mL of 100 mg L^{-1} Ni(II) solution and supplemented with 30 mg of the optimized Na-SH sorbent. The sealed flasks were placed in a shaking thermostat at 200 rpm for 30 min to allow adequate interaction between Ni(II) ions and the sorbent. After the contact period, the suspensions were filtered, and the remaining Ni(II) concentration in the filtrate was quantified by FAAS. The equilibrium adsorption capacity (q_e, mg g^{-1}) and removal efficiency (%R) were calculated using Eqs. (1) and (2).

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

$$\%R = \frac{C_i - C_e}{C_i} \times 100 \qquad (2)$$

3. Results and discussion

3.1. pH impact

Sorption studies were also conducted between pH 1-7 under optimum sorbent dose, initial metal ion concentration, contact time, and temperature to see the effects of changes in the pH on Ni(ll) removal using Na-SH from contaminated water. As evidenced in Figure 1, the transition of Ni (II) is not noticeable at pH ≤ 3.0 in Na-SH. This may be due to the fact that at a low pH, -COOH functional groups of Na-SH become protonated and converted into acidic form of sorbent (S-SH) with poor ion-exchange capacity as well as reduced sorptive ability. At the lower pH, H(I) ions and Ni(II) compete for sorption sites Na-SH. The sorption of H(I) is more favorable than that for Ni(II), decreasing the adsorption capacity of Na-SH. Interestingly, the Na-SH surface is negatively charged at a high pH region as it due to de protonation of Na-SH that favor the sorption of Ni(II). This may be the reason for faster sorption rate of Ni(II) species by the Na-SH used from crude water. The adsorption of Ni(II) was decreased gently when the pH was more than 6, similar to other metal ions reported before which attributed the result to colloidal precipitation of Ni(OH)2 in basic environment [28, 29]. The highest sorption was reached at pH 6. Therefore, pH 6 was selected as the optimum pH in further tests.

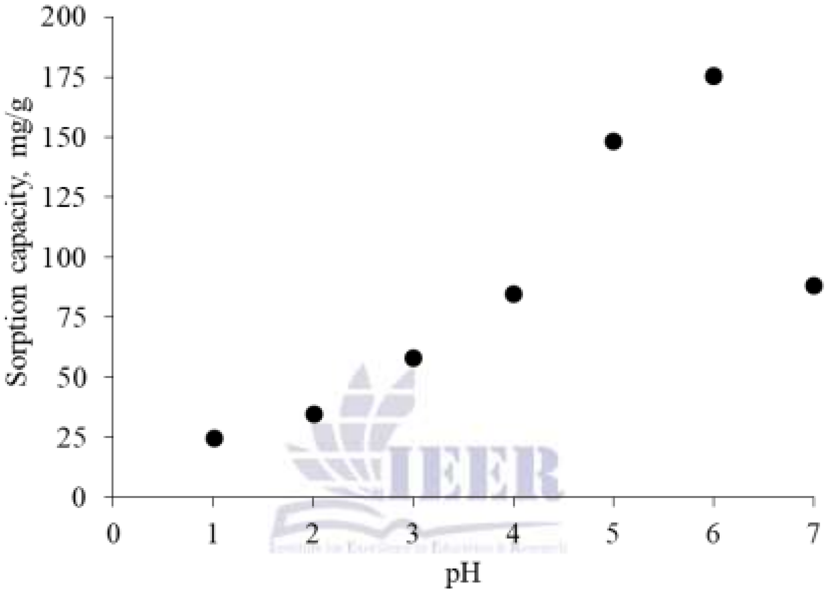

Figure 1: pH effect on Ni(II) adsorption.

3.2. Sorbent dose effect

Due to the fact that the ability of holding heavy metal ions by sorbents may be greatly influenced by excessive amounts. It is therefore important to determine the optimum sorbent concentration at which it can be able to remove maximum amount of metal ions from their contaminated water. Effect of Na-SH dose on the sorption behaviour of Ni(II) from the contaminated water was evaluated. The effect of dose 910- 90 mg (Na-SH)] at optimum initial Ni(II) concentration (100 mg L^{-1}/100 mL) at optimum pH value = 6, temperature=298 K and time = 30 min was monitored to verify influence of Na-SH in removing Ni(II) from contaminated water. This could be attributed to the prevailing greater surface sites for sorption with increased amount of Na-SH from 10 mg to 30 mg, and the consequent enhanced percentage uptake of Ni(II). However, the maximum sorption capacity decreases after an optimum Na-SH dosage (30 mg; i.e. 50-90 mg) has been reached (Figure 2). It could be suggested due to aggregation of Na-SH which left its surface sites unsaturated while the sorption process continued and then no active sorption site was available for get Ni(II). As the concentration of Na-SH increases, the competition for Ni(II) sorption among the Na-SH active sites is increasing; It has been reported that increase in concentration of Na-SH may also be attributable to decrease in the adsorption of Ni(II) [30,31]. Therefore, it is recommended that 30 mg Na-SH may be used for applying the present material in its industrial applications and all further Ni(II) sorption studies.

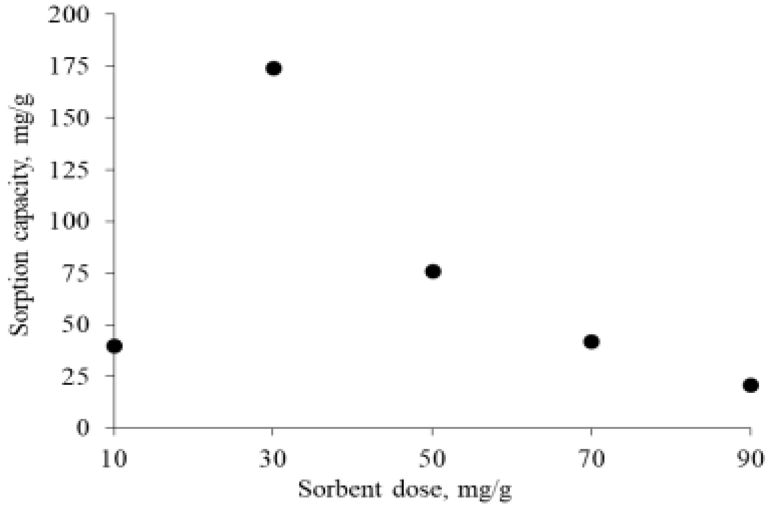

Figure 2: Na-SH dose effect on Ni(II) adsorption.

3.3. The impact of the initial concentration of metal ions

The sorption capacity of Na-SH was evaluated using initial Ni(II) concentrations ranging from 10 to 250 mg L^{-1} to elucidate the underlying sorption mechanism. An increase in the sorption capacity was observed as the initial Ni(II) concentration increased from 10 to 100 mg L^{-1}. The concentration of 100 mg L^{-1} was identified as the optimum level, at which the highest removal of Ni(II) ions was achieved. This trend can be attributed to the enhanced mass-transfer driving force at higher Ni(II) concentrations, which promotes more effective interaction between the sorbate and the active sites on the sorbent. Beyond this point, saturation of available binding sites likely limits further increases in sorption. This promoted the progress of Ni(II) diffusion from the bulk solution to Na-SH surface, which resulted in better sorption. However, the sorption capacity becomes constant at an optimum concentration of (100 mg L^{-1}) as shown in Figure 3, where no more vacant sites are accessible for Ni(II). This may be attributed to the fact that most of the Ni(II) ions produced during sorbate interact with Na(I) from the Na-SH, resulting an increase in Ni(II) concentration. However, in the fully saturated stage all exchangeable Na+ of Na-SH will be replaced by Ni(II) of sorbate; and Ni(II) will occupy Na-SH surface. It is found that the sorption capacity ultimately reached a stabilization state [32, 33].

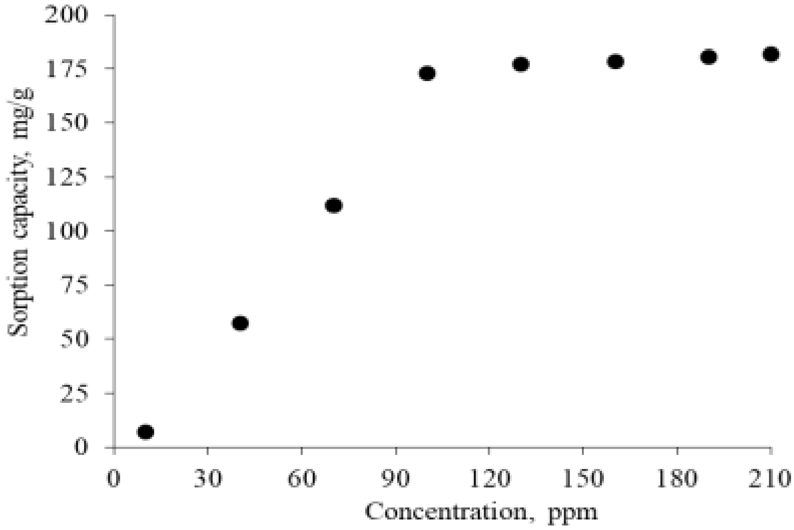

Figure 3: Concentration effect on Ni(II) adsorption.

3.4. Contact time's impact

To determine the sorption activity of Na-SH and to determine sorption kinetics for understanding sorption mechanism, contact time between sorbent and sorbate was studied. The sorption rate of Ni(II) on Na-SH were relatively faster at first (Figure 4), and the sorption capacity from polluted water increased or approached maximal values as early as the first 15 min. The optimum for the maximum equilibrium sorption capacity was 30 min and this indicates that the sorption equilibrium based on Ni(II) can be achieved within 30 min. In other words, the rate of sorption focused to saturation after 30 min. This is probably the result of the relatively high concentration of Ni(II) at the beginning of sorption process, and a large part of these adsorbate hydrated would have easily got to slightly low free energy while in contact with the surface of Na-SH holding many active sites could be loaded. But it was found that with increasing the sorption of Ni(II), the concentration of Ni(II) decreased, and therefore a high repulsive force formed between Ni(II) ions and aqueous solution. Consequently, Na-SH active effective sites reduced and there remained no Ni(II) to be absorbed. Ultimately, sorption rate fixed and retarded [8-10].

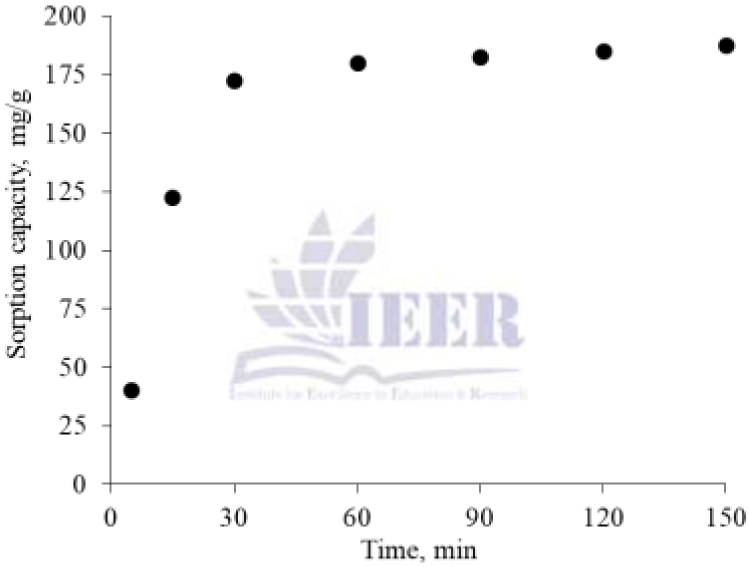

Figure 4: Time effect on Ni(II) adsorption.

3.5. The impact of temperature

In order to better understand the thermodynamic behavior of the adsorption process, temperature effect on the sorption capacity of Na-SH for Ni(II) removal from contaminated water was systematically investigated. As shown in Figure 5, the equilibrium adsorption of Ni(II) decreased gradually while temperature from 298 K to 338 K changed, The value for maximum sorption capacity was achieved at 298 K indicating that a low temperature is beneficial for Ni(II) uptake on Na-SH. This temperature variation of the sorption process reveals an exothermic nature of the adsorption process. At lower temperature, the Ni(II) ions lacks in kinetic energy and high opportunity to form stronger interaction with the functional groups and active binding sites on the surface of Na-SH. The lessened molecular mobility would promote the stability of surface complexes by an increased electrostatic attraction or ion-exchange reactions, according to the prevailing mechanism. As the temperature rises, however, several factors co-operatively reduce sorbate–sorbent interaction. They suggested that the increased kinetic energy due to the increasing temperature, increases the mobility and diffusion rate of Ni(II) ions in the solution which reduces their surface residence time on sorbent and thereby lessens complex formation. Furthermore, high temperature can partially break or loosen active sites for binding of Ni(II) which may have some influence on the total attraction of Na-SH to the metal ions. Thus, adsorption process can be exothermic in nature and it corroborates with general trend of thermodynamics that at higher temperature, heat-absorbing sorption is less favored. This is also supported by the thermodynamic parameters (*ΔG°*, *ΔH°* and *ΔS°*), which collectively confirm that Ni(II) sorption onto Na-SH was spontaneous and exothermic.

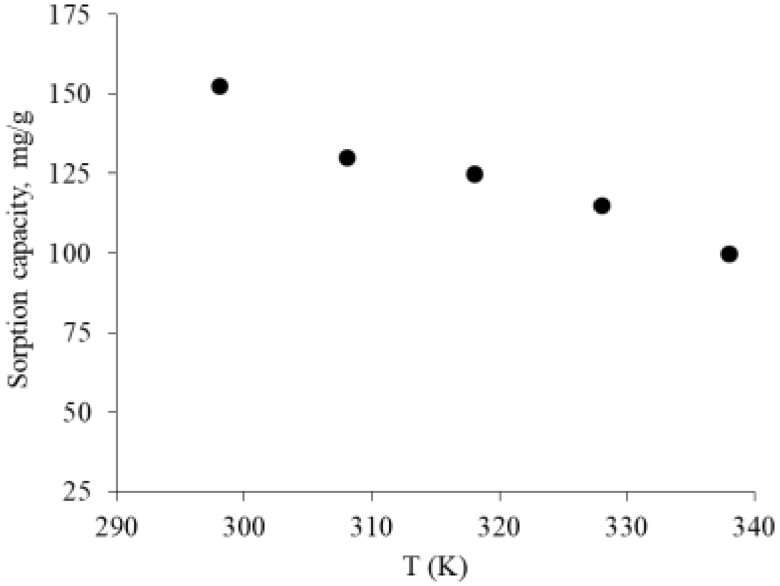

Figure 5: Temperature effect on Ni(II) adsorption.

4. Conclusion

After being esterified to succinylated SH, the hydrogel (SH) extracted from *S. spinosa* seeds were transformed into sodic form (Na-SH) which was then assessed for Ni(II) uptake from the polluted water. In 30 min, the Ni(II) sorption equilibrium was reached. The maximum Ni(II) uptake using Na-SH adsorbent was found to be 172.34 mg/g at Na-SH dosage of 20 mg, Ni(II) ions concentrations of 100 mg L^{-1}, contact time of 30 min, temperature of 298 K, and pH of 6. Furthermore, because the SH contains a Na-succinate moiety, it is therefore, Na-SH was discovered to be an ideal ion-exchanger for Ni(II) remediation from polluted water. In conclusion, the sorbent Na-SH showed promise as a super sorbent for heavy metal ion uptake; as a result, this material can also be used commercially to purify water.

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