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Research Article

The Sorption of Ni(II) by Grape Shell Ash from Aqueous Solution: Kinetic and Thermodynamic Studies

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Abstract

The sorption of Ni(II) onto grape shell ash (GSA) was studied by performing batch kinetic sorption experiments. The influences of major parameters in Nickel(II) ions sorption on GS such as initial pH, initial concentration of Ni(II) ions, the initial temperatures of solution, and contact time were investigated. The maximum increase in the rate of sorption of Ni(II) ions on GS was observed at an initial pH = 5, initial concentration of nickel 50 mg L⁻¹, temperature of solution (328 K), and $t = 90$ min. The rate constants and the equilibrium sorption capacities were calculated. The results indicate that the sorption process follows the second-order kinetics and the values of rate constants were found to be 0.224, 0.402, 0.193 and 0.123 min⁻¹ at 298, 308, 318, and 328 K, respectively. The values of correlation coefficients for the adsorption of Ni(II) on GSA from all the systems were found to be 0.999, and the values of predicted equilibrium sorption capacities showed good agreement with the experimental equilibrium uptake values. The thermodynamic parameters (ΔG° , ΔH° , and ΔS°) of the adsorption process were calculated, and these parameters showed that the adsorption process is spontaneous.

1. Introduction

The presence of heavy metals in the environment is of major concern because of their toxic nature and tendency for bioaccumulation in the food chain even in relatively low concentrations [1–5]. The discharge of water containing heavy metals causes critical pollution problems. Nickel (II) ion is one such heavy metal frequently encountered in wastewater streams from industries such as electroplating, battery manufacture, mineral processing, steam-electric power plants, paint formulation, and porcelain enameling [6–8]. In drinking water and for industrial wastewater, the tolerance limit of nickel is 0.01 mg L⁻¹ and 2.0 mg L⁻¹. Cancer of nose, bone, and lungs is created. Dermatitis (nickel itch) is the most frequent effect of exposure to nickel, such as coins and costume jewelry. Nickel carbonyl [Ni(CO)₄] has been estimated as lethal in humans at atmospheric exposures of 30 mg L⁻¹ for 30 min [3]. Acute Ni(II) poisoning causes dizziness, headache, nausea and vomiting, chest pain, dry cough and shortness of breath, rapid respiration, cyanosis, and extreme weakness [9–12]. These harmful effects of Ni(II) necessitate its removal from wastewaters before the release into streams. Several treatment methods such as membrane filtration, chemical precipitation, chemical oxidation/reduction, ion exchange, filtration, electrochemical treatment, solvent extraction, co-precipitation, and adsorption have been reported for the removal of metallic ions from water and wastewater [13]. Adsorptive removal is based on the ability of a porous adsorbent to selectively adsorb some specific compounds from the atmosphere or refinery streams. The compounds, which have a suitable size and shape, can be removed via adsorption. Based on the types of interactions between an adsorbate and a porous sorbent, the adsorption can be categorized as a physical or chemical one [14]. Various porous adsorbents such as activated carbons, zeolites, and mesoporous materials have been investigated for adsorptive removal of hazardous compounds.

A grape is a fruiting berry of the deciduous woody vines of the botanical genus *Vitis*. Grapes can be eaten raw or they can be used for making jam, juice, jelly, grape seed extract, raisins, vinegar, and grape seed oil. Italy ranks first in grape production in the world and Iran ranks eleventh in grape production, a potential of 3 million tons of grape are produced annually. The total area planted of grape is 310,000 hectare. This shows a high potential of grape shells produced during pruning step each year that have little value. This leads to a need to convert this by-product to useful, value added product, such as adsorbent. To our knowledge, no investigations have used grape shells as precursor to produce adsorbent and ash.

In this study, we attempt to utilize grape shell waste, an agricultural waste available in Iran as a sorbent to remove Ni ions from aqueous solution. This study mainly focuses on the effect of some environmental parameters such as solution pH, initial Ni(II) concentration, contact time, and temperature on the ability of Grape Shell ash (GSA) to biosorb Ni(II) ions from aqueous solutions and to determine the mechanism that govern Ni(II) ions removal as well as to find a suitable kinetic model for the adsorption process.

2. Experimental

2.1. Preparation of the Adsorbent

GS was obtained from one of the villages of Arak in Iran. Unmodified sorbent was washed several times with warm distilled water to remove impurities and was dried at 105°C for 24 h and cut into small pieces of sizes between 2.0 and 5.0 mm. The material was placed in a vertical stainless steel reactor and heated in a furnace at a rate of 10°C min⁻¹ from room temperature to 450°C. The black residue was cooled and sieved to get GS ash (GSA) with an average particle size of 0.125 mm and finally stored in air glass container.

2.2. Reagents

Solution of metal ions (nickel nitrate) was of analytical grade. Synthetic stock solution (1000 mg L⁻¹) of Ni(II) ions was prepared by dissolving the required quantity of Ni(NO₃)₂·6H₂O in double distilled water. Working solutions of the desired concentration were then prepared by successive dilution. All the solutions were made using deionized distilled water. pH adjustments were performed with HNO₃ and NaOH (Merck) solutions 0.1 N. All chemicals used in the experiments were reagent analytical grade.

2.3. Apparatus

The Ni ion concentration in the solutions was determined by Atomic Absorption Spectrometer (model AA 680 made of SHIMADZU) using a standard calibration curve. The pH was measured via the Swiss-made Metrohm 827 pH meter with a combined glass electrode.

2.4. Batch Adsorption Experiments

Batch experiments were conducted in order to study the effect of important parameters like the pH, contact time, and the initial ion concentration on the adsorptive removal of Ni ion using GSA. For the batch adsorption experiments, 50 mL solution of Ni(II) of initial concentration 10 mg L⁻¹ was contacted with 50 mg GSA. The contents were placed in a stirrer and gently agitated at 150 rpm. The solution was filtered, and the residual Ni concentration was analyzed. The effect of the initial pH onto the GSA was studied across a pH range of 2.0–5.0 in different time with a fixed adsorbent concentration. The effect of the initial concentration and contact time on the uptake of the Ni ions was conducted by varying the ion concentration from 5.0 to 50.0 mg L⁻¹ at different contact times (0.0–90.0 min). The percent of Ni(II)

$$R\% = \left[\frac{C_i - C_e}{C_i} \right] \times 100, (1)$$

$$q_e = \frac{C_i - C_e}{m} V, \quad (2) \quad \text{where } C_e \text{ and } C_i \text{ are the equilibrium and initial concentrations of Ni(II) (mg L}^{-1}\text{), respectively; } q_e \text{ is equilibrium Ni(II) concentration on adsorbent (mg g}^{-1}\text{); } V \text{ is the volume of Ni(II) solution (L); and } m \text{ is the mass of GSA sample used (g).}$$

removal (%R) and the amount of adsorbed Ni(II) at equilibrium, q_e , were calculated as follows:

3. Results and Discussion

3.1. Effects of Solution pH and Agitation Time

One of the main parameters affecting the metal adsorption from aqueous solution is the pH of the solution. The influence of solution pH and agitation time on the removal of Ni(II) by GSA was represented in Figure 1 and Table 1, for a period of 90 min. With increasing pH from 2 to 5, adsorption capacity of adsorbent increased from 7.2 to 9.03 mg g⁻¹.



Table 1: The maximum amount of Ni(II) sorbate as a function of pH at 20 min.

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Figure 1: The influence of solution pH and agitation time on the removal of Ni(II) by GSA (Ni(II) concentration, 10 mg L⁻¹; GSA dose, 1 g L⁻¹; Agitation time, 90 min).

In lower pH, a higher concentration of H⁺ ions present in the mixture competes with Ni(II) ions for the adsorption sites resulting in the reduced uptake of Ni(II). On the contrary, as the pH increases, the adsorbent surface becomes more and more negatively charged, and the adsorption of Ni(II) ions is more favorable. Figure 1 shows that the Ni(II) removal by GSA increases with increase in pH and the maximum of removal attains at 20 minutes.

3.2. Effect of Initial Concentration and Agitation Time

Figure 2 and Table 2 represent the removal of Ni(II) as a function of initial concentrations ranging from 5–50 mg L⁻¹ by GSA. The uptake efficiency is increased from 4.344 to 23.517 mg g⁻¹ with the increasing in initial Ni(II) concentration. This increase may be due to the ratio of the initial number of moles of Ni(II) to the available adsorption sites in adsorbent that is high at higher initial concentrations. Thus, at the same time, the time required for attaining the equilibrium state is highly independent on initial Ni(II) concentration. In Figure 2, the influence of contact time on the adsorption of Ni(II) on GSA is observed. According to Figure 2, the amount of Ni(II) adsorption onto GSA increases sharply up to the first 10 minutes and finally attain equilibrium where there is no further increase in the amount of adsorption. The uptake of adsorption of other adsorbents for the removal of Ni(II) from aqueous phase is given in Table 3 for comparison [15–18].



Table 2: The maximum amount of Ni(II) sorbate as a function of initial concentration of Ni(II) at 40 minutes.

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Table 3: Comparison of adsorption capacity of various adsorbents for the removal of Ni(II) from aqueous phase.

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Figure 2: The influence of initial concentration and agitation time on the removal of Ni(II) by GSA (pH = 5, 10 mg L⁻¹; GSA dose, 1 g L⁻¹; Agitation time, 90 min).

3.3. Adsorption Kinetic Study

Determination of uptake kinetics is important for the evaluation of a candidate adsorbent material. In order to estimate the uptake capacity of the sample in this study, three sorption kinetic models developed, pseudo-first-order, pseudo-second-order and Elovich equations by Lagergren [19], Ho and McKay [20], and Fatehi et al. [21], respectively, were used.

$$\log (q_e - q_t) = \log q_e - \frac{k_1}{2.303} t, (3)$$

$$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{t}{q_e}, \quad (4) \quad \text{where } q_e \text{ and } q_t \text{ are the amounts of Ni(II) adsorbed (mg g}^{-1}\text{) at equilibrium time and at time } t \text{ (min) and } k_1 \text{ and } k_2 \text{ is the rate constants of adsorption.}$$

The first and second order rate expressions given by (3) and (4) are respectively,

$$\text{The simplified form of Elovich's equation is shown in (5) is } q_t = \frac{1}{b} \ln (a \cdot b) + \frac{1}{b} \ln (t), (5) \quad \text{where } a \text{ is the initial adsorption rate (mg g}^{-1} \text{ min}^{-1}\text{), and the parameter } 1/b \text{ (mg g}^{-1}\text{) is related to the available sites for adsorption.}$$

Initially, the validity of the two models was checked by studying the kinetics under different initial temperatures of solution. Linear plots of $\log (q_e - q_t)$ and t/q_t versus t showed the applicability of the above equations (the first and second order) for GSA (Figures 3 and 4). The k_1 and k_2 values were calculated from the slopes of the linear plots and are presented in Table 4. The Elovich constants (a and b) computed from the plots of q_t versus $\ln t$ (Figure 5) are presented in Table 4. Correlation coefficients of the pseudo-first-order kinetic were found to be lower than 0.76, and the calculated q_e is not equal to experimental q_e , suggesting the insufficiency of pseudo-first-order model to fit the kinetic data for the initial concentrations examined. The reason for these differences in the q_e values is that there is a time lag, possibly due to a boundary layer or external resistance controlling at the beginning of the sorption process [22]. In most cases of the literature, the pseudo-first-order model does not fit the kinetic data well for the whole range of contact time, and generally underestimate the q_e values [23, 24]. The pseudo-second order model is based on the sorption capacity of the solid phase. Contrary to other well established models, it predicts the behavior over the whole range of studies and it is in agreement with a chemisorption mechanism being the rate-controlling step [22].



Table 4: The pseudo-first-order, pseudo-second-order, and Elovich's equation kinetic parameters for Ni(II) sorption onto GSA at different temperatures of solution.

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Figure 3: The pseudo-first-order plots for the adsorption of Ni(II) by GSA. (Ni(II) concentration, 10 mg L⁻¹; GSA dose, 1 g L⁻¹; pH = 6).

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Figure 4: The pseudo second order plots for the adsorption of Ni(II) by GSA. (Ni(II) concentration, 10 mg L⁻¹; GSA dose, 1 g L⁻¹; pH = 6).

Figure 5: Elovich's equation plots for the adsorption of Ni(II) by GSA. (Ni(II) concentration, 10 mg L⁻¹, GSA dose, 1 g L⁻¹; pH = 6).

The values of correlation coefficients for the adsorption of Ni(II) on GSA from all the systems were found to be 0.999 for pseudo-second-order kinetic model, and the values of predicted equilibrium sorption capacities showed good agreement with the experimental equilibrium uptake values. Based on correlation coefficients and q_e values calculated, it is evident that the adsorption of Ni(II) can be best described by the pseudo-second-order kinetic model (Table 3).

Results of Table 3 also showed that the k_2 values decrease as temperature rises, confirming that the rate of biosorption is faster at lower temperatures, which is probably due to a decrease in the interactions between Ni(II) ions and GSA as the biosorbent. This behavior is a characteristic of exothermic reactions.

The R^2 values were in the range of 0.484 to 0.926 for the various temperatures ranging from 298 K to 328 K for Elovich's equation. The q_e values calculated from the Elovich equation disagreed with the experimental q_e values. It was observed that the Elovich model constants, namely, a (mg g⁻¹ min⁻¹) and b (mg g⁻¹), decreased with temperature.

3.4. Adsorption Mechanism

To confirm whether Ni ions adsorption on GSA is dominated by intraparticle diffusion (Weber and Morris), the empirical data were analyzed using the intraparticle diffusion model [25] $q_t = k_i t^{0.5} + I$, (6) where q_t (mg g⁻¹) is the uptake of Ni ions at time t , k_i (mg g⁻¹ min⁻¹) is the intraparticle diffusion rate constant, and I (mg g⁻¹) indicates the thickness of the boundary layer, that is, larger values of I suggest greater boundary layer effects. The calculated data for the adsorption of Ni(II) ions on to GSA at different temperature applied to diffusion model is shown in Figure 6, and the intraparticle diffusion constants are given in Table 5. No plots of model passed through the origin, showing that intraparticle diffusion was not sole rate-controlling step although it was involved in the process. This emphasized that adsorption of Ni(II) ions was a two or more steps process [26].

Table 5: The intraparticle diffusion kinetic parameters for Ni(II) sorption onto GSA at different temperatures of solution.

Figure 6: The intraparticle diffusion plots for the adsorption of Ni(II) by GSA. (Ni(II) concentration, 10 mg L⁻¹, GSA dose, 1 g L⁻¹; pH = 6).

3.5. Adsorption Thermodynamic Study

The thermodynamic parameters, Gibbs free energy (ΔG°), enthalpy (ΔH°), and entropy (ΔS°) have an important role to determine spontaneity and heat change for the

$$k_D = \frac{q_e}{C_e}, \quad (7)$$

$$\Delta G = -RT \ln k_D, \quad (8)$$

adsorption process. The thermodynamic parameters (ΔG° , ΔH° and ΔS°) were calculated using the following equations: $\ln k_D = \frac{\Delta S}{R} - \frac{\Delta H}{RT}$, (9) where k_D is the distribution coefficient of the adsorbate and q_e and C_e are as defined previously. R is the universal gas constant (8.314 J mol⁻¹ K⁻¹) and T is temperature (K) [27]. ΔH° and ΔS° can be obtained from the slope and intercept of the plot $\ln k_D$ against $1/T$, respectively (Figure 7). ΔG° was obtained using (8) for different temperatures. The data in Table 6 showed that the standard free energy changes (ΔG°) are negative (−8.5078, −8.0296, and −7.9642 kJ mol⁻¹ at 298, 308, and 318 K, resp.), indicating spontaneous adsorption of Ni(II) ions on GSA for the temperature range studied (298, 308 and 318 K). The negative ΔH° value (−16.6762 kJ mol⁻¹) showed the endothermic nature of the sorption process. Since diffusion is an exothermic process, it would be expected that increased solution temperature would result in decrease uptake of Ni(II) ions from aqueous solution. Also, the negative value of ΔS° (−0.0276 kJ mol⁻¹ K⁻¹) may be related to the decrease randomness.

Table 6: Thermodynamic parameters for adsorption of Ni(II) onto GSA.

Figure 7: Plot of $\ln K_D$ against temperature ($1/T$) for the adsorption of Ni(II) by GSA.

4. Conclusion

The sorption characteristics of Grape Shell were studied for Ni(II). The results indicated that this adsorbent may be used as an inexpensive and effective material for the removal of Ni(II) from aqueous solutions but with a low efficiency. The sorption process was affected by experimental conditions such as pH, initial concentration of Ni(II) ions, the initial temperatures of solution, and contact time. Analysis of the kinetic data showed that the kinetics of Ni(II) adsorption using Grape Shell ash as an adsorbent for different values of initial temperatures of solution is explained by the second-order-kinetic model. The calculated thermodynamic parameters determined the spontaneous and endothermic nature of the Ni(II) biosorption process onto GSA.

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