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Removal of Fe(II) from aqueous solution using pomegranate peel carbon: equilibrium and kinetic studies

Masoud Rohani Moghadam^{1*}, Navid Nasirizadeh², Zienab Dashti³ and Esmail Babanezhad⁴

Abstract

Background: Pomegranate peel carbon was tested for its ability to adsorb iron(II) ions from solutions with alterations in the parameters such as pH, contact time, sorbent weight, metal concentration, and temperature.

Results: The adsorption process was analyzed on the basis of pseudo-first-order, pseudo-second-order kinetic models and also by Freundlich and Langmuir isotherm models. The maximum adsorption capacity for pomegranate peel activated carbon as calculated by Langmuir model was found to be 18.52 mg g⁻¹ at pH 6.0 and 1 g L⁻¹ of biosorbent concentration at 29°C.

Conclusions: The ability of new sorbent is compared with other biomass waste materials. Thermodynamic parameters such as the changes of free energy, enthalpy, and entropy were also evaluated. The results indicated that the sorption of iron(II) ions onto the pomegranate peel carbon was an exothermic process that could be well fitted with the Langmuir adsorption model and pseudo-first-order kinetic model.

Keywords: Biosorption, Iron(II) removal, Pomegranate peel carbon

Background

Punica granatum L. (Punicaceae), commonly called pomegranate, is one of the most popular fruits in the world due to its pleasant taste, high nutritional value, and many medical features. In Iran, pomegranate production is over 700,000 tons/year that is mainly cultivated in central part of the country. It consists of edible part, seeds, and peel. The pomegranate peel constitutes 5% to 15% of its total weight [1]. The extracted dye from the pomegranate peel had been used as natural colorant for textiles from ancient times. But nowadays, with the growth of synthetic dyes, pomegranate peel is discarded as a pollutant residue that can be used as a low cost and renewable source of biomass. Recently, some studies have been done using pomegranate peel to remove Cr(VI) [2], Ni(II) [3], Pb(II) [4], Cu(II) [5], and Congo red [6] from water samples.

Fe(II) is needed in plant metabolism, but when it presents in excess, it can become extremely toxic. Since

several decades along with the growth of industrial activities, contamination of the environment with wastewaters including heavy metals has become a major problem [7]. Iron ion as an important corrosive product is one of the substances that is commonly found in fluids of many industries. Conventional techniques aimed at removing heavy metals usually include chemical precipitation, ion exchange, membrane process, crystallization, and electrochemical treatment [8]. Although many investigations have been conducted so far about the ability of some biomass or waste materials to remove Fe(III) [9-18], only a few investigators have studied Fe(II) biosorption using crab shell [19], bark of a tree [20], waste coir fiber [21], wooden charcoal [22], and pine bark [23].

In this study, the ability of pomegranate peel carbon to remove Fe(II) from aqueous solutions has been assessed in a batch process. The effects of different parameters such as contact time of biosorbent and aqueous solution, pH of the aqueous solution, initial metal ion concentration, and different biomass amounts have been investigated. The biosorption isotherm and thermodynamic parameters were deduced from biosorption measurements.

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Methods

Preparation of adsorbent using pomegranate peel waste

According to [5], the waste peels of pomegranate fruit were first washed with double-distilled water and dried in an oven at 70°C overnight. The dried pomegranate peels were grounded, and this material was then thermally activated at 500°C in a furnace for 1 h in the absence of air. After activation, the ash was removed by washing it with HCl solution (0.1 M) and distilled water and then dried again in an oven at 90°C overnight. The particle size of BSS 150 to 200 (0.075 to 0.104 mm) was obtained by sieving, and this product was kept in a desiccator for further use. Porosity (%), density (kg m^{-3}), moisture content (wt.%), and ash content (wt.%) were 60, 584.6, 6.79, and 9.98, respectively.

Adsorption studies

The adsorption of iron on pomegranate peel carbon adsorbent was studied by batch method. A known volume of Fe(II) solutions with different initial concentrations was taken in a 50-mL conical flask and agitated in a mechanical shaker at 120 rpm with a known dose of adsorbent for a specified contact time. After equilibration, samples were filtered using Whatman 42 filter paper (Maidstone, Kent, UK), and the concentration of iron was determined according to spectrophotometric standard method [24]. The data obtained in the batch mode was used to calculate the equilibrium metal adsorptive quantity using the following expression:

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

where q_e is the amount of heavy metal ion adsorption per unit weight of the biomass (mg g^{-1}); V , the volume of solution treated (L); C_0 , the initial concentration of metal ion (mg L^{-1}); C_e , the equilibrium metal ion concentration (mg L^{-1}); and m , the biomass (g).

Results and discussion

Effect of pH

pH has a direct influence on the biosorption mechanism and uptake of metal by the adsorbent. To understand the effect of pH, the adsorption of Fe(II) as a function of pH in the range of 2.5 to 7.5 was measured, and the result was shown in Figure 1. There was an increase in the biosorption capacity of the biomass with the increase in pH from 2.5 to 5.0 and was maximum in the pH range of 5.0 to 6.0. Then, it decreased when pH reached 7.5. This fact can be explained by the competition between the surface charge and solution chemistry as two important factors. In highly acidic pH, adsorbent surface is protonated and act as a positive charge. Deprotonation occurs when pH increases and causes negative charges

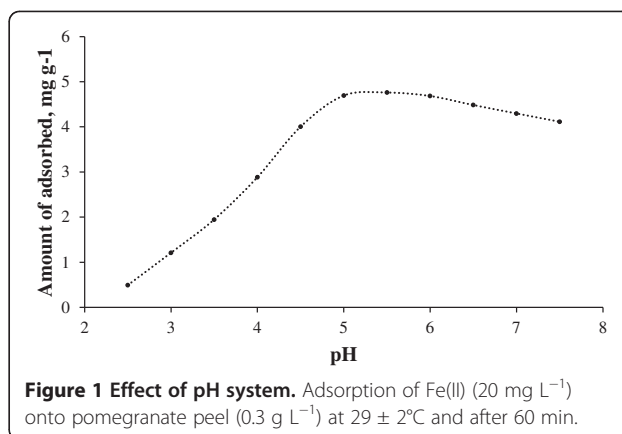


Figure 1 Effect of pH system. Adsorption of Fe(II) (20 mg L^{-1}) onto pomegranate peel (0.3 g L^{-1}) at $29 \pm 2^\circ\text{C}$ and after 60 min.

on the surface to appear, and these negative charges attract Fe(II) ions. The pH can also affect the solution chemistry. In acidic pHs, Fe(II) remains with the positive charge, but in alkali pHs, the hydroxide ions (OH^-) causes precipitation of Fe(II) in the form of $\text{Fe}(\text{OH})_2$.

Effect of contact time

To know the kinetics of the adsorption process and equilibration time on pomegranate peel carbon, the effect of contact time on Fe(II) biosorption was studied, and the results were shown in Figure 2. According to Figure 2, the adsorption quantity of Fe(II) ion increases as the contact time increases. The equilibration was nearly reached after 40 min. Therefore, equilibration period of 40 min was selected for all further experiments. The time profiles are single, smooth, and continuous curves leading to saturation, suggesting the possible monolayer coverage of Fe(II) on the surface of the adsorbent.

Effect of initial metal ion concentration

The metal uptake mechanism was particularly dependent on the initial Fe(II) concentration. Biosorption experiments with pomegranate peel carbon in equilibrium time were conducted for solutions containing 1 to 100 mg L^{-1} Fe(II) ion. Figure 3 shows the effect of Fe(II) initial concentration

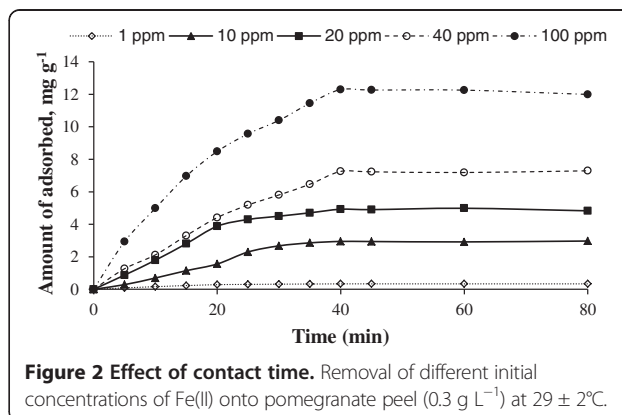


Figure 2 Effect of contact time. Removal of different initial concentrations of Fe(II) onto pomegranate peel (0.3 g L^{-1}) at $29 \pm 2^\circ\text{C}$.

on the removal amount of Fe(II) in percentage. It is clear that with increasing initial concentration, the amount of metal removal decreases. At low concentrations, Fe(II) is adsorbed to specific sites, while with the increase of Fe(II) concentration, the specific sites are saturated and the exchange sites are occupied.

Effect of biosorbent concentration

The effect of biosorbent concentration as one of the parameters that strongly affects the adsorption capacity is shown in Figure 4. It can be observed that with the fixed Fe(II) concentration, the amount of removed Fe(II) ions increases with the increase of the adsorbent weight. The increase in Fe(II) removal percentage by increasing the adsorbent dose is due to greater availability of the exchange sites or surface area.

Kinetic modeling

One of the most important characteristics in evaluating adsorption efficiency is kinetics. In order to clarify the biosorption kinetics of Fe(II) ions onto pomegranate peel carbon, the kinetic models, Lagergren's pseudo-first-order [25] and pseudo-second-order models [26], were used. For kinetic studies, different concentrations of Fe(II) ranging from 1 to 40 mg and biosorbent concentration of 0.3 g L⁻¹ were used at optimum condition of pH (6.0) and agitation time (40 min).

The Lagergren model as a pseudo-first-order rate equation is obtained under the ideal assumption of a totally homogenous adsorption surface and is given as follows:

$$\log(q_e - q_t) = \log q_e - \frac{K_1 t}{2.303},$$

where q_e and q_t are the amounts of adsorbed metal ion (mg g⁻¹) at equilibrium and at time t , respectively. K_1 is the Lagergren rate constant relating to adsorption energy

(L min⁻¹). The values of K_1 and q_e that are presented in Table 1 are calculated from the slope and intercept of $\log(q_e - q_t)$ versus t .

The pseudo-second-order kinetic model of McKay and Ho can be expressed as follows:

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

where q_e and q_t are the amounts of iron adsorbed (mg g⁻¹) at equilibrium and at time t (min), respectively, and k_s (g mg⁻¹ min⁻¹) is the rate constant of pseudo-second-order kinetics. The values of q_e and K_s that are presented in Table 1 are calculated from the slope and intercept of t / q_t versus t .

As can be seen from Table 1, the theoretical q_e (cal) values in the case of the pseudo-first-order model agree well with the experimental uptake values, q_e (exp), whereas the pseudo-second-order model does not give reasonable values. Further, the correlation coefficient (R^2) for the pseudo-first-order kinetic model was better than the pseudo-second-order model. Thus, the pseudo-first-order kinetic model was suggested to describe the kinetic behavior of Fe(II) adsorption onto pomegranate peel carbon.

Adsorption isotherms

The isotherm model is a mathematical model to describe the distribution of metal ions between the solid and liquid phases that are in contact. The adsorption of Fe(II) at optimum pH (6.0) and agitation time (40 min) was carried out at different concentrations ranging from 1 to 100 mg L⁻¹ and 0.1 to 1 g L⁻¹ for ions and biosorbent, respectively. Adsorption isotherm can be used to describe how the solute interacts with the adsorbent and so is critical in optimizing the use of adsorbent. In addition, it can be used for column design purposes. In this research, adsorption isotherm study was carried out

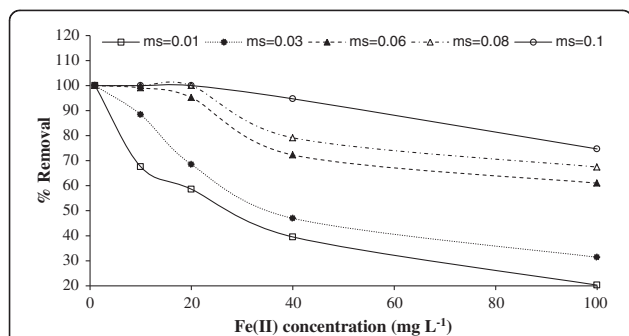


Figure 3 Effect of initial metal ion concentration. Removal of different initial concentrations of Fe(II) onto different dosages of pomegranate peel at 29 ± 2°C.

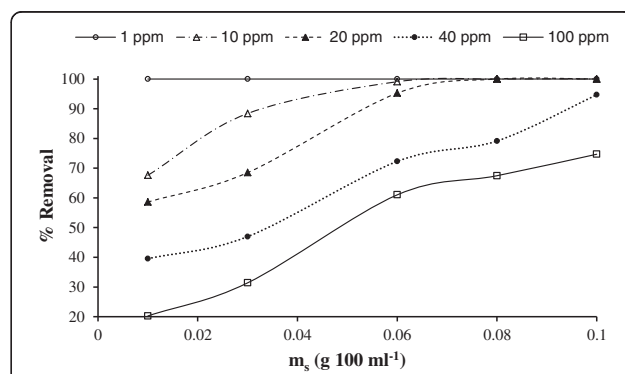


Figure 4 Effect of biosorbent concentration. Removal of different initial concentrations of Fe(II) onto different dosages of pomegranate peel at 29 ± 2°C.

Table 1 Comparison of pseudo-first-order and pseudo-second-order model parameters, and calculated and experimental amount adsorbed values

| Concentration (ppm) | First-order kinetic model | | | | Second-order kinetic model | | |
|---------------------|-----------------------------------|------------------------------------|------------------------------|-------|------------------------------------|---|-------|
| | q_e (exp) (mg g ⁻¹) | q_e (calc) (mg g ⁻¹) | K_1 (L min ⁻¹) | R^2 | q_e (calc) (mg g ⁻¹) | K_s (g mg ⁻¹ min ⁻¹) | R^2 |
| 1 | 0.333 | 0.203 | 0.0714 | 0.993 | 0.372 | 0.4773 | 0.996 |
| 10 | 2.947 | 3.467 | 0.0737 | 0.997 | 7.246 | 0.0026 | 0.893 |
| 20 | 4.927 | 5.212 | 0.0990 | 0.999 | 6.369 | 0.0108 | 0.984 |
| 40 | 6.261 | 6.982 | 0.1313 | 0.998 | 7.752 | 0.0158 | 0.988 |

q_e (calc), calculated amount adsorbed value; q_e (exp), experimental amount adsorbed value.

on two well-known isotherm models for the single-solute systems, i.e., Langmuir and Freundlich.

Langmuir isotherm

The Langmuir model [27] is one of the first theoretical models. It assumes that biosorption occurs at specific homogenous active sites of the sorbent. In addition, the energy of adsorption is constant, and there is no migration of adsorbate molecules in the surface plane. This model, which is used successfully in many monolayer biosorption processes, is given by the equation:

$$q_e = \frac{q_m K_L C_e}{1 + K_L C_e},$$

where q_m (mg g⁻¹) is the monolayer adsorption saturation capacity; K_L (L mg⁻¹), Langmuir constant relating to adsorption energy; q_e (mg g⁻¹), the amount of metal ion bound to per gram of the biomass at equilibrium; and C_e , the residual (equilibrium) metal ion concentration left in the solution after binding. The q_m and K_L can be determined from C_e / q_e versus the C_e plot which gives a straight line of slope $1 / q_m$ and intercept $1 / K_L \cdot q_m$. As shown in Figure 5, R^2 values are greater than 0.9983 which indicate that the biosorption of Fe(II) onto pomegranate peel carbon follows the Langmuir model. The maximum adsorption capacity for pomegranate peel activated carbon

as calculated by Langmuir model was found to be 18.52 mg g⁻¹ at pH 6.0 and 1 g L⁻¹ of biosorbent concentration at 29°C.

The Freundlich isotherm

The Freundlich isotherm [28] model is based on sorption heterogeneous energetic distribution of active sites accompanied by interactions between adsorbed molecules. This model is expressed by the following:

$$q_e = K_F C_e^{1/n},$$

where K_F (mg^{1 - 1/n} g⁻¹ L^{1/n}) is the adsorption capacity and n is the adsorption intensity of metal ions on the sorbent that can be determined from the linear plot of $\log(q_e)$ versus $\log(C_e)$. The linear Freundlich isotherm plots are presented in Figure 6. Values of the correlation coefficients show that the Freundlich isotherm is poorly appropriate to characterize for the adsorption of the Fe(II) ions onto pomegranate peel carbon.

Table 2 gives the maximum capacity and required equilibration time of different waste adsorbents for the removal of Fe(II) from aqueous solutions. It can be seen that the values are different for different materials. This depends on the physical nature and chemical composition of the waste substances. The pomegranate carbon peel has relatively short equilibration time and also has

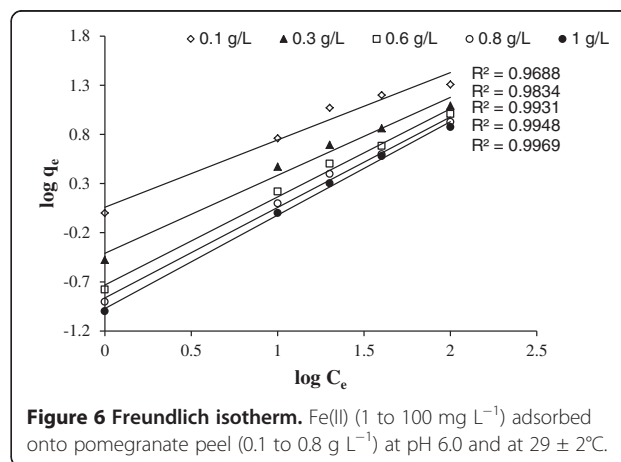
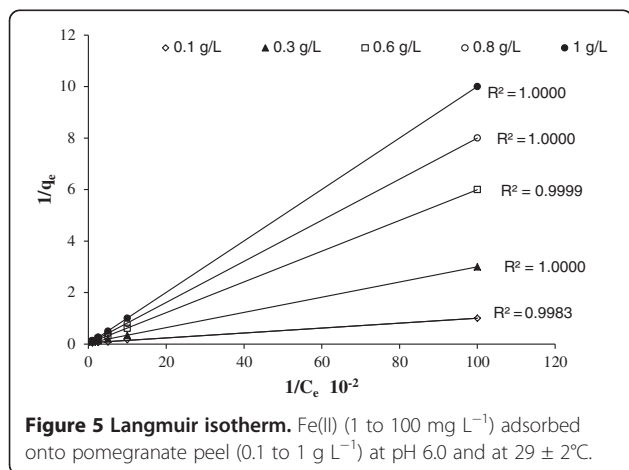


Table 2 Comparison of adsorption capacities and equilibrium times of different waste adsorbents for Fe(II) removal

| Waste material | Equilibrium time (min) | Adsorption capacity (mg g ⁻¹) | Reference |
|--|------------------------|---|-----------|
| Crab shells (raw) | 840 | 1.08 | [19] |
| Bark of wild jack tree (carbon active) | 90 | 0.2570 | [20] |
| Bark of wild jambul tree (carbon active) | 90 | 0.1978 | [20] |
| Coir fiber (raw) | 120 | 2.84 | [21] |
| Oxidized coir fiber (raw) | 120 | 7.49 | [21] |
| Wooden charcoal (raw) | 150 to 180 | 1.88 | [22] |
| Calabrian pine bark (raw) | 30 | 2.03 | [23] |
| Pomegranate peel (carbon active) | 40 | 18.52 | This work |

the best capacity compared to all of the other waste materials used for removing the Fe(II).

Thermodynamic parameters

In order to confirm the nature of adsorption process, the important thermodynamic parameters such as the standard Gibbs free energy, ΔG° , enthalpy, ΔH° , and entropy, ΔS° , changes were calculated according to the following equations [5]:

$$\Delta G = -RT \ln K$$

$$\ln \frac{K_2}{K_1} = \frac{\Delta H}{R} \left(\frac{1}{T_1} - \frac{1}{T_2} \right)$$

$$\Delta G = \Delta H - T \Delta S,$$

where R is the universal gas constant; T , the absolute temperature in Kelvin; and K , the equilibrium constant that can be calculated by multiplying the molar concentration of the solvent (55.5 mol L⁻¹) to the Langmuir constant 'b' [3]. For the determination of the thermodynamic parameters, experiments in two different temperatures of 29°C and 50°C were done. As shown in Figure 7, adsorption decreases with increasing temperature, suggesting that the process is exothermic in nature.

Thermodynamic parameters obtained for the adsorption of Fe(II) ions onto pomegranate peel carbon are given in Table 3. As shown in the table, the negative

value of ΔG° confirms the feasibility of the process and the spontaneous nature of the sorption. The values of ΔH° were negative, indicating that the sorption reaction is exothermic. The negative value of ΔS° shows the decreasing randomness at the solid/liquid interface during the adsorption.

Experimental

Reagents and instruments

All reagents used were of analytical reagent grades and were purchased from the Merck company (Darmstadt, Germany). All the solutions were prepared with double-distilled water with zero levels of dissolved oxygen. Iron stock solution was prepared by (NH₄)₂Fe(SO₄)₂·6H₂O. The stock solution was diluted further to obtain the standard solutions. *Ortho*-phenanthroline solution was prepared by dissolving 1 g of 1,10-phenanthroline monohydrate in water and diluting to 1,000 mL. Buffer solution (pH = 6) was prepared by dissolving appropriate amounts of ammonium acetate in acetic acid. Iron concentration was obtained from absorbance measurement by a Jenway spectrophotometer model 6505 (Jenway, England, UK). Measurements of pH were made with a Jenway 3510 pH meter using a combined glass electrode.

Conclusion

In this study, pomegranate peel carbon biosorbent was applied successfully for the biosorption of Fe(II) from aqueous solution. The kinetic studies proved that the first-order kinetic was the best applicable model. The isotherm equilibrium studies confirmed that the Langmuir form and generalized models were the highest fitted models for the adsorption process. The maximum adsorption potential of pomegranate peel adsorbent for Fe(II) removal was ca. 18.5 mg g⁻¹. Thermodynamic parameters showed that

Table 3 Thermodynamic parameters for adsorption of Fe(II) ions onto pomegranate peel carbon at 29°C

| K | ΔG° (J mol ⁻¹) | ΔH° (J mol ⁻¹) | ΔS° (J mol ⁻¹ K ⁻¹) |
|-------|---|---|---|
| 1.288 | -635.57 | -1,083.54 | -1.48 |

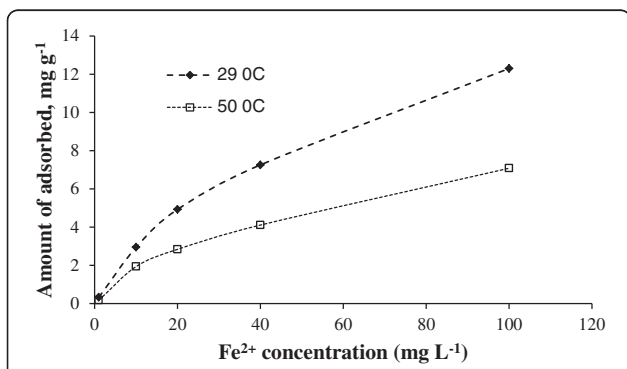


Figure 7 Removal efficiency of Fe(II) by 0.3 g L⁻¹ of pomegranate peel at 29 ± 2°C and 50 ± 2°C.

the adsorption process was exothermic and spontaneous. The comparison of this study with the previous studies shows that the new biosorbent has more ability than the other waste biomasses for removing Fe(II).

Competing interests

The authors declare that they have no competing interests.

Authors' contributions

MRM, ZD, and NN carried out the experiments, and EB participated in the calculations. All authors read and approved the final manuscript.

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