

original article

Decolorization of reactive red 198 by adsorption onto ZnCl₂ activated pistachio hull wastes

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ABSTRACT

Aims: In the present study, activated carbon obtained from pistachio hull waste ash was used as an adsorbent for the successful removal of reactive red 198 dye from aqueous solutions.

Materials and Methods: The effect of various operating parameters such as initial concentration of dye (20-200 mg/L), contact time (5-270 min), adsorbent dosage (0.1-5 g/L) and pH (2-12) was investigated. Furthermore, equilibrium isotherms were used to identify the possible mechanism of the adsorption process.

Results: Maximum adsorption capacity of the adsorbent was 253.67 mg/g at pH 2, initial concentration of 80 mg/L and 25°C, when 95.13% of the dye was removed. Furthermore, the equilibrium data were also fitted to the Langmuir and Freundlich equilibrium isotherm models. It was found that the data fitted to Freundlich ($R^2 = 0.9289$) better than Langmuir ($R^2 = 0.8839$) model.

Conclusion: It was concluded that the activated carbon obtained from pistachio hull waste ash can be used for dyes removal from water and wastewater.

Key words: Adsorption, pistachio hull wastes, reactive dyes

INTRODUCTION

The textile industry consumes large volumes of water in different wet processes; therefore, very large amounts of textile wastewater, which is heavily charged with unconsumed dyes and other chemical are produced.^[1] About 10-20% of the total dyestuff used in the dyeing process is released into the environment.^[2]

Dyes are one of the most hazardous chemical compound classes found in industrial effluents and need to be treated

since their presence in water bodies reduces light penetration, precluding the photosynthesis of aqueous flora.^[3,4] They are also aesthetically objectionable for drinking and other purposes^[5] and can cause allergy, dermatitis, skin irritation^[6] and also provoke cancer^[7] and mutation in humans.^[8]

Inappropriate treatment and disposal of dye-contaminated wastewaters from textile, dyeing and related industries have provoked serious environmental concerns all over the world.^[9-12] Removal of dye in wastewater has been made by physical, physico-chemical, biological and/or chemical processes.^[13-15] The conventional treatment process of textile effluents involves numerous stages due to the characteristics of the production process.^[16] Conventional treatment involves a process of coagulation/flocculation. This is a versatile process, which can be used alone or combined with biological treatments as a way of removing suspended solids and organic material as well as promoting the extensive removal of dyes from textile industry

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effluents.^[17,18] However, this approach presents the disadvantage of generating a large volume of sludge. This sludge is rich in dyes as well as other substances used in the textile process. This is a problem, as the waste must be discarded properly to avoid environmental contamination.^[16] Furthermore, biological and enzymatic treatment,^[11,19-22] ozone treatment,^[11,23,24] nanoparticles,^[25] chemical oxidation and photocatalytic processes,^[12,26,27] photochemical and sonochemical processes,^[28] and membrane processes^[9,11,29] were used for removal of dye from textile effluents. However, some of these methods are limited due to their high operational costs and problems.

The most efficient procedure for removal of synthetic dyes from industrial effluents is the adsorption procedure, because the dye species are transferred from the water effluent to a solid phase, diminishing the effluent volume to a minimum. Subsequently, the adsorbent can be regenerated or kept in a dry place without direct contact with the environment.^[4] Also, adsorption has proven to be a reliable treatment methodology due to its low capital investment cost, simplicity of design, ease of operation and insensitivity to toxic substances, but its application is limited by the high price of some adsorbents and the large amounts of wastewater normally involved. Activated carbon,^[30,31] mesoporous carbon,^[32] clay minerals,^[33] hydrotalcite,^[34] biopolymers such as chitosan beads^[35] and quaternary chitosan^[36] and agricultural by-products^[37,38] are a few of the adsorptive materials that have been tested for the treatment of wastewaters.

In order to reduce preparation cost, the use of low cost starting materials (industrial or agricultural residues) for activated carbon preparation has emerged as a potential alternative.^[39] Besides the cost reduction, the conversion of agriculture waste in low cost adsorbents also added value to this residues and solve the problem of biomass disposal. Agricultural waste-based carbon has the advantage of exhibiting low ash content, reasonable hardness and high surface area and/or adequate porous structures.^[29,39]

The choice of activated carbon precursor largely depends on its availability, cost and purity, but the manufacturing process and intended applications of the product are also important considerations.^[40] Therefore evaluation of biomass is getting increased attention in all over the world as it is renewable, widely available, cheap and environmental friendly.^[41]

Iran is one of the major pistachio (*Pistacia vera*) producer and exporter country. Pistachio production is nearly 200,000 t/year.^[41] Thus, pistachio wastes generated from pistachio processing has a major economic importance and exhibits a high affinity for dyes. Therefore, in this study, adsorption of reactive red 198 (RR198) from aqueous solutions on activated carbon prepared from pistachio hull wastes was studied. The effects of different parameters including initial pH, adsorbent dosage, RR198 concentration and contact time were studied. In addition, the sorption isotherm was explored to describe the experimental data.

MATERIALS AND METHODS

Chemicals and reagents

RR198 is an anionic dye with molecular weight of 968.21 g/mol and $\lambda_{\text{max}} = 530$ nm. The RR198 ($\text{C}_{27}\text{H}_{18}\text{ClN}_7\text{Na}_4\text{O}_{15}\text{S}_5$) used in this work was the analytical grade (Merck, Germany). The chemical formula of RR198 is shown in Figure 1. For treatment experiments, the dye solutions with concentrations in the range of 20-200 mg/L were prepared by successive dilution of the stock solution (1000 mg/L) with distilled water. All other chemicals used in this study were of analytical grade.

Adsorbent preparation

Pistachio hull wastes (*Pistacia vera*) used in the batch experiments were collected from lands near to Rafsanjan city (30°23' N, 56°00' E) of Kerman province in the southeastern part of Iran. This natural wastes were firstly washed with distilled water to remove impurity such as sand and leaves and soluble and colored components, dried at 110°C for 2 h, crushed in a domestic grinder and sieved to obtain particle size in the range of 60-200 mesh. Then a mixture of 250 g of powdered adsorbent, 110 g of ZnCl_2 and 110 g of water was kept overnight and then the mixture was calcinated at 350°C. Then, chemically prepared activated carbon was washed with water and dried. The activated carbon was then used in adsorption experiments. No other chemical or physical treatments were used prior to adsorption experiments.

Dye removal experiments

Dye removal experiments with the pistachio hull waste ash were carried out as batch tests in 250 mL flasks under magnetic stirring. Each test consisted of preparing a 100 mL of dye solution with a desired initial concentration and pH by diluting the stock dye solution with distilled water and transferring it into the beaker on the magnetic stirrer. The pH of the solution was adjusted using 1 N hydrochloric acid or sodium hydroxide solutions. A known mass of pistachio hull waste ash (adsorbent dosage) was then added to the solution and the obtained suspension was immediately stirred for a pre-defined time. After the desired contact time, the samples were withdrawn from mixture by using a micropipette and centrifuged for 5 min at 5000 rpm. After centrifuged, supernatants were analyzed for the determination of the final concentration of RR198 by using

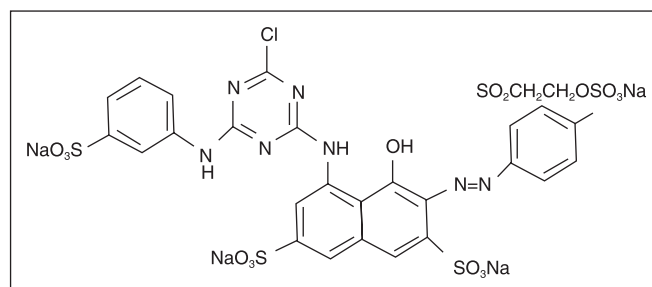


Figure 1: Structure of reactive red 198

an UV-VIS spectrophotometer (T80 PG Instruments Ltd) set at a wave-length of 518 nm (maximum of wavelength absorbed). Then the amount of RR198 adsorbed, q_e (mg/g), was obtained as follows:

$$q_e = \frac{(C_0 - C_e)V}{M} \quad (1)$$

Where, C_0 and C_e are the initial and equilibrium liquid phase concentration of RR198 (mg/L), respectively. V is the volume of the solution (L) and M is the amount of adsorbent used (g).

To express the percent of dye removal, the following equation was used:

$$\% = \frac{(C_0 - C_f)}{C_0} \cdot 100 \quad (2)$$

Where, C_0 and C_f represent the initial and final (after adsorption) dye concentrations, respectively. All tests were performed in duplicate to insure the reproducibility of the results; the mean of the two measurements is reported. All experiments were performed at room temperature ($\sim 25^\circ\text{C}$). The investigated ranges of the experimental variables were as follows: RR198 dye concentration (20, 30, 50, 70, 80, 100, 150 and 200 mg/L), pH of solution (2-12), adsorbent dosage (0.1, 0.3, 0.5, 0.7, 1, 2, 3, 4 and 5 g/L) and mixing time (5-270 min).

RESULTS

Effect of initial pH

In our experimental study, for study the effect of pH on the RR198 adsorption capacities of the activated carbon from pistachio hull wastes, adsorption experiments were conducted at varying pH (pH 2-12) with 80 mg/L fixed initial dye concentrations and adsorbent dosage 0.3 g/L for 60 min and the results are shown in Figure 2. As can be seen from Figure 2, a decrease in pH from 12 to 2 caused a significant increase in the amount of RR198 adsorbed by adsorbent. The maximum adsorption capacity of the adsorbent was 253.67 mg/g at pH 2, initial concentration of 80 mg/L and 25°C , when 95.13% of the dye was removed.

Effect of adsorbent dosage

In order to determine the effect of adsorbent dosage on adsorption, 0.1-5 g/L adsorbent were used for adsorption experiments at fixed initial pH (pH 2), initial dye concentration (80 mg/L) and temperature (25°C) for 60 min.

As it can be seen from Figures 3 and 4 the percentage of RR198 removal steeply increases with the adsorbent loading up to 0.3 g/L. The maximum adsorption efficiency of RR198 onto adsorbent was found to be 96.23% (256.6 mg/g) at adsorbent concentration of 0.3 g/L.

Effect of contact time

The effects of contact time on adsorption were studied at 25°C , initial dye concentration (80 mg/L) and 0.3 g/L

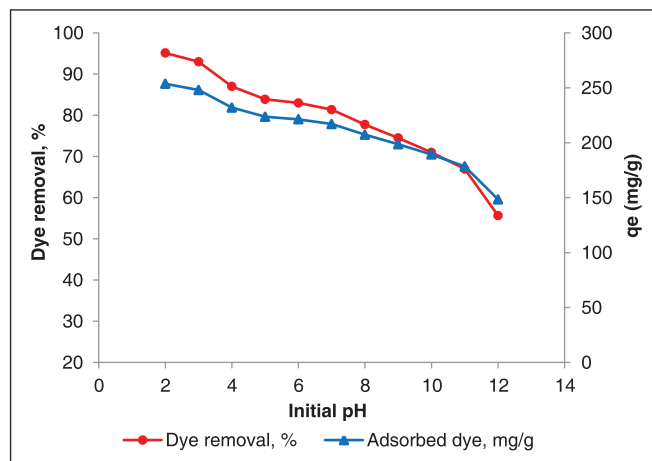


Figure 2: Effect of initial pH on reactive red 198 adsorption onto activated carbon from pistachio hull wastes

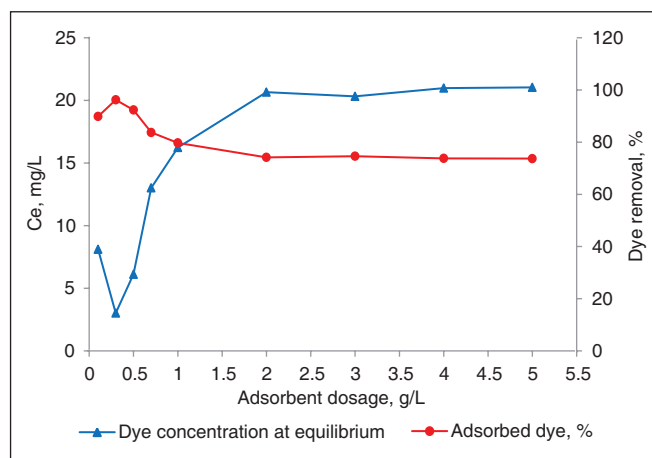


Figure 3: Effect of adsorbent dosage on reactive red 198 adsorption onto activated carbon from pistachio hull wastes

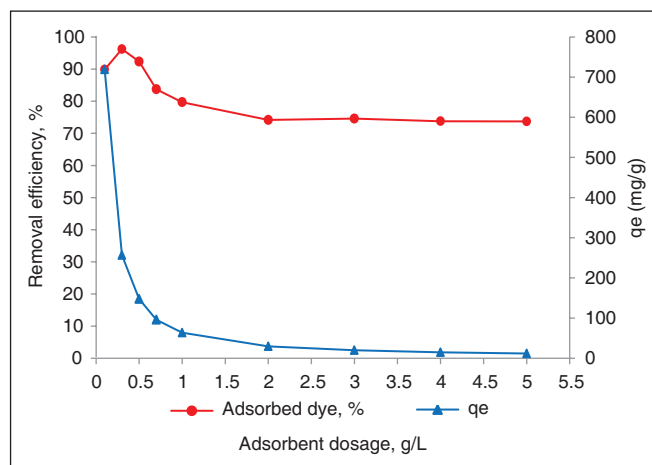


Figure 4: Effect of adsorbent dosage on reactive red 198 adsorption onto activated carbon from pistachio hull wastes

adsorbent dosage for 5-270 min. Figure 5 shows the effect of contact time on the adsorption capacity and percent removal of RR198 dye onto the activated carbon from pistachio hull wastes at different initial concentrations. According to Figure 5 the RR198 was rapidly adsorbed in the first 20 min (73.63%) and then the adsorption rate decreased gradually from 20 min to 270 min and finally reached to equilibrium in about 120 min. Furthermore, the maximum efficiency (96.19%) for removal of RR198 was obtained at contact time 60 min (256.5 mg/g).

Effect of initial RR198 dye concentration

In this study, the effects of initial dye concentration on adsorption were studied at 25°C and 0.3 g/L adsorbent concentrations for 60 min. As can be seen from Figure 6, when the initial dye concentration is increased, the amounts of adsorbed dye also increase, so the removal of dye depends on the concentration of the dye. For example, when the initial RR198 concentration increases from 20 mg/L to 200 mg/L,

the equilibrium sorption capacities of activated carbon from pistachio hull wastes increase from 59.76 (89.64%) to 629.97 mg/g (94.5%).

Adsorption isotherms

Isotherms study can describe how an adsorbate interacts with adsorbent. The isotherm provides a relationship between the concentration of dye in solution and the amount of dye adsorbed on the solid phase when both phases are in equilibrium. Figures 7 and 8 shows the equilibrium isotherms for the adsorption of RR198 dye onto pistachio hull waste and the equilibrium adsorption data were analyzed by using the Langmuir and Freundlich isotherm models. The Langmuir isotherm model is valid for monolayer adsorption onto surface containing finite number of identical sorption sites, which is presented by the following equation:

$$q_e = \frac{q_m K_l C_e}{1 + K_l C_e} \tag{3}$$

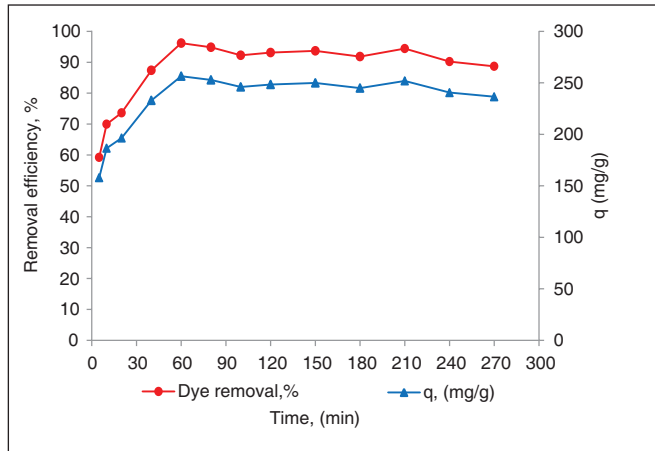


Figure 5: Effect of contact time for the adsorption of reactive red 198 onto activated carbon from pistachio hull wastes

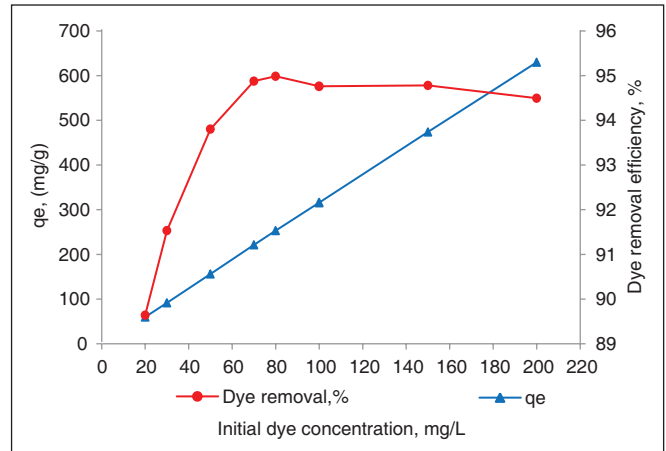


Figure 6: Effect of initial reactive red 198 dye concentration for its adsorption onto activated carbon from pistachio hull wastes

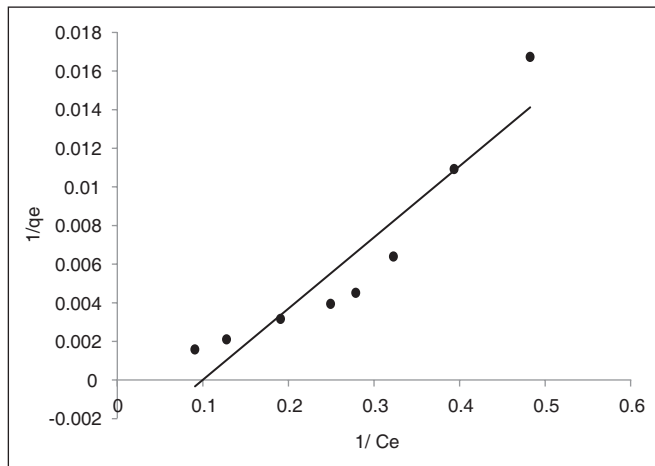


Figure 7: Langmuir isotherm for sorption of reactive red 198 onto activated carbon from pistachio hull wastes ($R^2 = 0.8839$)

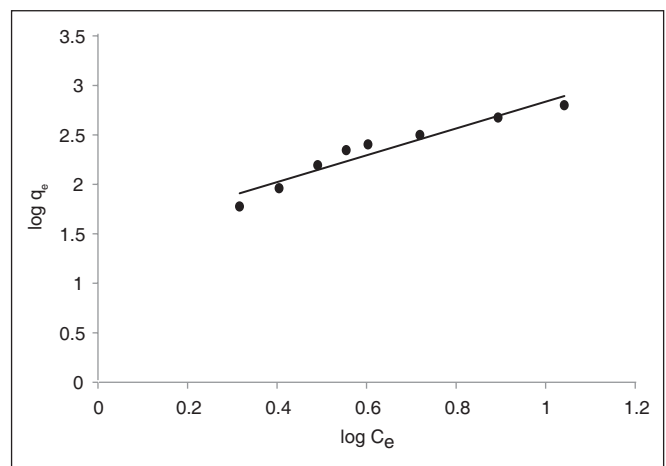


Figure 8: Freundlich isotherm for sorption of reactive red 198 onto activated carbon from pistachio hull wastes ($R^2 = 0.9289$)

Where, q_e is the amount of metal adsorbed per specific amount of adsorbent (mg/g), C_e is equilibrium concentration of the solution (mg/L) and q_m is the maximum amount of RR198 dye required to form a monolayer (mg/g). The Langmuir equation can be rearranged to linear form for the convenience of plotting and determining the Langmuir constants (K_L) and maximum monolayer adsorption capacity of adsorbent (q_m). The values of q_m and K_L can be determined from the linear plot of $1/q_e$ versus $1/C_e$:

$$\frac{1}{q_e} = \frac{1}{q_m} + \frac{1}{q_m K_L} \frac{1}{C_e} \quad (4)$$

The Freundlich equation is purely empirical based on sorption on heterogeneous surface, which is commonly described by the following equation:

$$q_e = K_f C_e^{\frac{1}{n}} \quad (5)$$

where, K_f and $1/n$ are the Freundlich constants related to adsorption capacity and adsorption intensity, respectively. The Freundlich equilibrium constants evaluated from the intercept and the slope, respectively of the linear plot of $\log q_e$ versus $\log C_e$ based on experimental data. The Freundlich equation can be linearized in logarithmic form for the determination of the Freundlich constants as shown below:

$$\log q_e = \log K_f + \frac{1}{n} \log C_e \quad (6)$$

The isotherms based on the experimental data and the parameters obtained from nonlinear regression by both models are shown in Figures 7 and 8. Also, all parameters are listed in

DISCUSSION

Effect of initial pH

It is known that the solution pH can affect the surface charge of the adsorbent, the degree of ionization of the different pollutants, the dissociation of functional groups on the active sites of the adsorbent as well as the structure of the dye molecule.^[42] the solution pH would affect both aqueous chemistry and surface binding-sites of the adsorbent. Hence, the solution pH is an important parameter during the dye adsorption process

As can be seen from Figure 2, a decrease in pH from 12 to 2 caused a significant increase in the amount of RR198 adsorbed by adsorbent. On the other hand, the adsorption of RR198 dye onto activated carbon from pistachio hull wastes is intimately dependent on solution pH. It could be seen that the uptake of RR198 increased with decreasing pH from 12 to 2 until equilibrium obtained. The maximum adsorption capacity of the adsorbent was 253.67 mg/g at pH 2, initial concentration of 80 mg/L and 25°C, when 95.13% of the dye was removed. Similar results were reported by other

researchers.^[13,43-45] at acidic conditions, binding sites of the adsorbent would be closely associated with the hydrogen ions, which act as bridging ligands between the adsorbent surface and the dye molecule.^[46] The lower pH values can be suitable for the adsorption of reactive dye.^[47-50]

Effect of adsorbent dosage

The adsorbent concentration is an important parameter because this determines the capacity of the adsorbent (activated carbon from pistachio hull waste) for a given initial RR198 concentration. As it can be seen from Figures 3 and 4 the percentage of RR198 removal steeply increases with the adsorbent loading up to 0.3 g/L. This result can be explained by the fact that the sorption sites remain unsaturated during the sorption, whereas the number of sites available for sorption site increases by increasing the adsorbent dose. There was a non-significant increase in the percentage removal of RR198 when the adsorbent concentration increases beyond the 0.3 g/L and even in contrast with, a few of decrease in removal efficiency was determined. This suggests that after a certain dose of adsorbent, the maximum adsorption is attained and hence the amount of pollutants remains constant even with further addition of dose of adsorbent.^[51] Also, when the adsorbent dosage increased, percentage removal of RR198 also increased (up to 0.3 g/L adsorbent), but amount of adsorbed RR198 of per gram adsorbent decreased due to the fact that fixed dye concentration (80 mg/L) led to unsaturated active site on adsorbent surface and increase in the adsorbent concentrations caused particle aggregation.^[52] A similar result was reported by Gulnaz *et al.*^[43]

Effect of contact time

The contact time is one of the most important parameters for practical application. As it can be seen from Figure 5, the adsorption capacity and percent removal of RR198 dye onto the adsorbent drastically increase during the initial adsorption stage and then continue to increase at a relatively slow speed with contact time until a state of equilibrium is attained after 120 min. In general, the removal rate of sorbate (pollutant) is rapid initially, but it gradually decreases with time until it reaches equilibrium. This phenomenon is attributed to the fact that a large number of vacant surface sites are available for adsorption at the initial stage and after a lapse of time, the remaining vacant surface sites are difficult to be occupied due to repulsive forces between the solute molecules on the solid and bulk phases. A similar finding was reported by Cengiz and Cavas,^[53] and Gulnaz *et al.*^[43]

Effect of initial RR198 dye concentration

The initial concentration provides an important driving force to overcome all mass transfer resistance of the dye between the aqueous and solid phases.^[47,54] As can be seen from Figure 6, when the initial dye concentration is increased, the amounts of adsorbed dye also increase, so the removal of dye depends on the concentration of the dye. Similar trends have

also been observed for Methylene blue dye sorption onto bamboo-based activated carbon,^[55] cotton waste^[56] and fly ash.^[57] This increase in the proportion of removed dye may be probably due to equilibrium shift during sorption process. Similar results have also been recorded for adsorption of Congo red from aqueous solution onto calcium-rich fly ash,^[58] RR198 removal from aqueous solutions by *Potamogeton crispus*^[43] and 2,4-D herbicide removal by single-walled carbon nanotubes.^[59]

The initial dye concentrations provide an important driving force to overcome the mass transfer resistance of the dye between the aqueous phases and the solid phases, so increasing initial concentrations would enhance the adsorption capacity of dye. These results is in accordance with obtained findings by other researchers.^[53,54]

Adsorption isotherms

The R^2 values of Langmuir and Freundlich models [Table 1 and Figures 7 and 8] are 0.8839 and 0.9289, respectively, indicating that the Freundlich model is more suitable for describing the adsorption equilibrium of RR198 dye onto activated carbon obtained from pistachio hull wastes. In the Langmuir model, it is assumed that the site energy for adsorption is the same for all surface sites and does not depend on degree of coverage and that the largest capacity corresponds to only one monolayer. These assumptions are not valid for most adsorbents because, for example, activated carbon has a wide range of pore size that continues to adsorb organics as the concentration increases. In reality, the site energy of adsorption varies widely for most adsorbents because adsorbents, such as activated carbon, are very heterogeneous and the site energy varies considerably with surface coverage. The Freundlich equation is used to describe isotherm data for heterogeneous adsorbents (varying site energies) much better than the Langmuir equation.^[60]

CONCLUSION

From the present study, it can be seen that the activated carbon from pistachio hull waste can be used effectively for the removal of the RR198 dye from aqueous solutions. The percentage eliminated was found to depend on the amount of adsorbent, the initial concentration of the dye and the dye-adsorbent contact time. The activated carbon from pistachio hull wastes was able to remove up to 95% of RR198 dye from solutions whose initial concentration varied

between 20 mg/L and 200 mg/L. The adsorption of RR198 dye on activated carbon from pistachio hull wastes has been described by the Langmuir and Freundlich isotherm. It was found that the data fitted to Freundlich ($R^2 = 0.9289$) better than Langmuir ($R^2 = 0.8839$) model. The removal of the dye from aqueous solutions is induced by adsorption on surface sites of the solid for low RR198 dye concentration while both adsorption and internal exchange take place for high concentrations.

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Table 1: Isotherm parameters for adsorption of RR198 onto activated carbon from pistachio hull wastes at 25 °C

Langmuir isotherm			Freundlich isotherm		
q_m (mg/g)	k_L (L/mg)	R^2	k_f	n	R^2
271.62	0.1	0.8839	30.22	0.74	0.9289

RR198: Reactive red 198

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