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Pomegranate seed powder as a new biosorbent of reactive red 198 dye from aqueous solutions: adsorption equilibrium and kinetic studies

M. T. Ghaneian · B. Jamshidi · M. Dehvari ·
M. Amrollahi

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Abstract Dyes are complex organic materials which are important causes of environmental contamination. The purpose of this study was evaluation of the adsorption equilibrium and kinetics for bioadsorption of reactive red 198 dye from aqueous solutions by pomegranate seed powder. The effects of amount of adsorbent, initial dye concentration, and retention time were evaluated. Adsorption experiments were conducted under acidic conditions (pH 3). Pomegranate seeds were washed with distilled water, boiled for 2 h, dried in an oven, and milled, and the powder was passed through standard ASTM sieves (40–100 mesh). Experimental results were analyzed by use of the Langmuir and Freundlich isotherm models and the pseudo-first-order and pseudo-second-order kinetic models. According to the results, adsorption capacity (removal efficiency, %) for 0.2 g/100 cm^3 adsorbent and initial dye concentrations 25 and 50 mg/l was 10.95 mg/g (87.64 %) and 17.75 mg/g (71 %), respectively. Results showed that increasing the amount of adsorbent and the retention time led to increased efficiency of dye removal, and that increasing the initial dye concentration led to reduced efficiency of dye removal. Maximum adsorption occurred after contact for 30 min. The experimental results were in good agreement with the Freundlich isotherm, and adsorption followed pseudo-second-order kinetics. On the basis of these results, this biosorbent enables effective removal of reactive red 198 dye.

Keywords Reactive red 198 dye · Pomegranate seed powder · Adsorption equilibrium and kinetics · Natural biosorbent

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M. T. Ghaneian · B. Jamshidi · M. Dehvari (✉) · M. Amrollahi
Department of Environmental Health, Faculty of Public Health, Shahid Sadoughi University of Medical Sciences, Yazd, Iran
e-mail: mahboobehdehvari@yahoo.com

Introduction

Dyes are important causes of environmental contamination. An indication of the scale of the problem is that 2 % of dyes produced annually are discharged in effluent from manufacturing operations, and 10 % of dyes are discharged by the textile industry [1]. On the basis of the color index, more than 10,000 different dyes are currently available worldwide [2]. Disposal of colored water degrades the environment of receiving waters by affecting the photosynthetic activity of aquatic life, because of reduced light penetration; it may also be toxic to aquatic life [3, 4], because of the presence of metals and chlorides [5]. Dyes also cause problems because they may be mutagenic and carcinogenic; they can also cause severe damage to humans, for example dysfunction of the kidneys, reproductive system, brain, and central nervous system [3, 4].

Reactive dyes usually contain azo-based chromophores. Because of favorable characteristics which include simple application techniques and low energy consumption, they are used extensively in the textile industry [5, 6]. Because dyes are stable, recalcitrant, carcinogenic, and toxic [2], their removal is very important [5, 6]. Biological, physical, and chemical processes have been reported for removal of dyes from wastewater [2, 7, 8]. Each of these has specific advantages and disadvantages [2]. A wide range of conventional treatment techniques, for example chemical coagulation, activated sludge treatment, carbon adsorption, and photodegradation, have been investigated for removal of dyes [9, 10]. Dyes can be effectively removed by adsorption [11]; advantages include low initial cost, simplicity of design, ease of operation, and insensitivity to toxic substances [12]. Dye adsorption is a process of transfer of dye molecules from the bulk solution phase to the surface and/or interface of the adsorbent. Most conventional adsorption plants use activated carbon [5].

Both chemical and thermal regeneration of carbon are expensive, impractical on a large scale, produce additional effluent, and result substantial loss of adsorbent [13]. Thus, many researchers have focused their efforts on optimizing adsorption by development of novel low-cost adsorbents with high adsorptive capacity [2]. The abundance and availability of natural materials, biosorbents, and waste materials from agriculture make them good sources of raw material for activated carbon [2, 12, 14]. Application of waste materials is gradually becoming of great concern, because these wastes are unused resources and can cause serious disposal problems. For better use of inexpensive and abundant agricultural waste, it has been investigated as a low-cost adsorbent, owing to its relatively high fixed carbon content and porous structure [15]. Studies have revealed that a variety of agricultural wastes, for example pommel (*Citrus grandees*) peel [16], sunflower seed shells [17], and guava (*Psidiumgua java*) leaf powder [18], are natural biosorbents.

Pomegranate (*Punica granatum*), a small tree originating in the orient, belongs to the *Punicaceae* family [19]. It is native from Iran to northern India and widely cultivated throughout Iran, India, the drier parts of Southeast Asia, Afghanistan, and Mediterranean countries, and to some extent in the USA and tropical Africa. It is one of the most valuable fruits and in grown on a commercial scale in Iran.

Pomegranate seeds, a by-product of pomegranate juice production, are inexpensive [12, 20, 21]. In this study, pomegranate seed powder was used as a new, low-cost adsorbent for removal of reactive red 198 (RR198) dye from aqueous solutions. The effect of different conditions, for example amount of adsorbent, contact time, and initial dye concentration were investigated. Finally, isotherm and kinetics data for the adsorption of RR198 dye by pomegranate seed powder were evaluated.

Experimental

Chemicals

RR198 dye, produced by Dystar, was obtained from Yazdbaf textile industry and used without further purification. The characteristics and chemical structure of the dye are given in Table 1 and Fig. 1 [2, 5]. Other chemicals were of analytical grade and purchased from Merck.

Preparation and characterization of biosorbent

Pomegranate seeds were washed with distilled water, boiled for 2 h, then dried in an oven to constant weight. The material was then milled by electric grinder and passed through American Society for Testing and Materials (ASTM) standard sieves (40–100 mesh) [12].

To investigate the properties of the adsorbent particles and to study their surface morphology before and after adsorption, particle texture was observed by scanning electron microscopy (SEM) by use of a KYKY-EM3200 (China) operated at 25 kV accelerating potential.

Adsorption experiments

To determine the optimum pH for adsorption, the effect of initial pH 3, 5, 7, and 9 was investigated (results not shown). The results showed that adsorption of RR198 dye on pomegranate seed powder was maximum at initial pH 3; for this reason all experiments were conducted at pH 3. The initial pH of solutions was adjusted by addition of H_3PO_4 (0.033 M); pH was monitored by use of an Mi 151 meter. According to Wang et al. [22] adsorption of textile and food dyes is maximum (100 %) under strongly acidic conditions.

In this research the effect of such conditions as initial dye concentration (25 and 50 mg/l), amount of adsorbent (0.05–0.5 g/100 ml), and contact time (10, 20, 30, 60, 90, 120 min, and 24 h) was investigated at pH 3 and 25 °C. Dye samples were prepared by dissolving a known quantity of the dye in distilled water as stock solution (1000 mg/l); this solution was diluted to the required initial concentrations. Batch experiments for removal of RR198 were conducted in 250-ml conical flasks containing 100 ml solution, and samples were mixed by use of a mechanical shaker (GFL 137; Innova, UK) at a constant speed of 150 rpm. To separate the adsorbents from the aqueous solutions; all samples were filtered [12] and dye concentrations

Table 1 Basic properties of RR198

Commercial name	Class	C.I. number	λ_{max} (nm)	Molar mass (g/mol)	Company
Remazol red 133	Azo	18221	518	984.2	Dystar

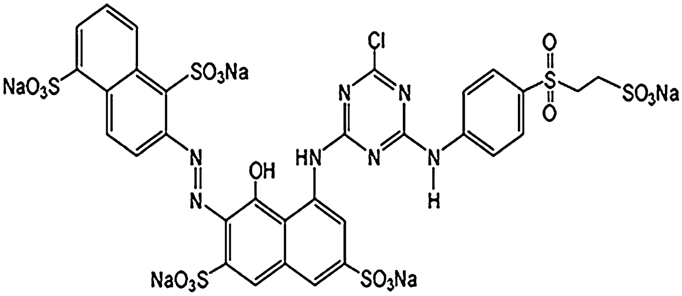

Fig. 1 Molecular structure of RR198 dye

remaining in solution were measured by UV–visible spectrophotometry (Optima SP-3000 Plus; Japan). Dye concentration was calculated by measurement of absorbance at λ_{max} (518 nm) use of a calibration curve constructed after measurement of the absorbance of known concentrations. Efficiency of removal of the dye was calculated by use of the equation:

$$\%\ \text{dye adsorption} = \frac{C_0 - C_t}{C_0} \times 100, \tag{1}$$

where C_0 and C_t (mg/l) are the initial and residual dye concentrations, respectively, at any time [12]. The amount of adsorption at equilibrium (adsorption capacity), q_e, was calculated by use of the equation:

$$q_e = \frac{(C_0 - C_e)V}{W}, \tag{2}$$

where q_e (mg/g) is the equilibrium adsorption capacity, and C_0 and C_e (mg/l) are, respectively, the liquid-phase concentrations of the dye under the initial and equilibrium conditions. V (l) is the volume of solution and W is the mass of dry adsorbent used (g) [12, 13].

Adsorption isotherms and kinetics

The adsorption isotherm is the relationship between the amount of the dye adsorbed and its concentration in the equilibrium solution. Adsorption isotherms are basic requirements for design of adsorption systems [12, 23]. To determine adsorption isotherms, two dye solutions (25 and 50 mg/l) were agitated with a known amount of adsorbent (0.05–0.5 g/100 cm^3) at fixed pH until equilibrium was reached (24 h). The results were then investigated by use of the Langmuir and Freundlich adsorption isotherms.

The Langmuir equation describes adsorption on homogeneous surfaces with negligible interaction between adsorbed molecules. This assumption is not valid for natural adsorbents, which are chemically heterogeneous because of their variety of functional groups [24]. The model takes the linear form:

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

where q_e is the amount adsorbed (mg/g), K_L is the Langmuir adsorption constant (l/mg), q_m is Langmuir constant related to maximum adsorption capacity (mg/g), and C_e is the dye concentration at equilibrium (mg/l) [2, 12].

The essential characteristic of the Langmuir isotherm is the separation factor or equilibrium term, R_L, defined by:

$$R_L = \frac{1}{1 + K_L C_0}, \tag{4}$$

where C_0 is the initial dye concentration and K_L is the Langmuir constant. R_L values indicate whether the isotherm is linear ($R_L = 1$), irreversible ($R_L = 0$), favorable ($0 < R_L < 1$), or unfavorable ($R_L > 1$) [2, 25].

The Freundlich isotherm models adsorption on a heterogeneous surface with a non-uniform distribution of heat of adsorption over the surface. It can be described in the linear form by use of the equation:

$$\log q_e = \log k_F + \frac{1}{n} \log C_e, \tag{5}$$

where n and k_F (l/mg) are isotherm constants indicating, respectively, the intensity and capacity of adsorption. The constants n and k_f were obtained from a plot of log q_e versus log C_e, for which the slope is $1/n$ and the intercept ln k_f [2, 12].

For kinetic studies, 100 ml dye solution of known concentration (25 and 50 mg/l) and initial pH 3 was placed in a 250-ml conical flask with the required amount of adsorbent and agitated in the shaker at 25 °C. Samples were withdrawn after different times (10–120 min). Different models can be used to express the mechanism of adsorption of a dye by a natural biosorbent. In this research we investigated pseudo-first-order and pseudo-second-order kinetic models.

A form of the pseudo-first-order model was described by Lagergren in the form:

$$\log(q_e - q_t) = \log q_e - \frac{K_f}{2.303} t, \tag{6}$$

where q_e and q_t (mg/g) are to the amount of dye adsorbed at equilibrium and at time t, respectively, and K_f is the pseudo-first-order rate constant (min^{-1}).

Pseudo-second-order kinetics expressed in a linear form as:

$$\frac{t}{q_t} = \frac{1}{K_2 q_e^2} + \frac{1}{q_e t}, \tag{7}$$

where the q_e is the equilibrium adsorption capacity and K_2 (g/mg h) the second-order rate constant. These can be determined from the slope and intercept of a plot of t/q_t versus t [13].

Results and discussion

Characterization of the biosorbent

SEM images of pomegranate seed powder before and after adsorption are shown in Fig. 2a, b. It is apparent pomegranate seed powder has an irregular surface structure, a situation which favors adsorption of RR198 by different parts of the biosorbent. It is clear these samples do not have well-defined pore structures.

Effect of amount of adsorbent

The effect of amount of adsorbent (0.05–0.5 g/100 ml) on adsorption of the dye by pomegranate seed powder at pH 3 is depicted in Fig. 3. Initial dye concentrations were 25 and 50 mg/l and the shaker speed was 150 rpm. The results showed that the amount of dye adsorbed per unit mass of adsorbent (q_e) decreased with increasing amount of adsorbent. On increasing the amount of adsorbent from 0.05 to 0.5 g for a dye concentration of 25 mg/l, adsorption capacity changed from 23.12 to 6.21 mg/g; for 50 mg/l, adsorption capacity changed from 28.14 to 10.37 mg/g. According to these results, for the optimum amount of adsorbent (0.2 g/100 cm^3) and 25 and 50 mg/l initial dye concentrations, adsorption capacity after 24 h was 10.95 and 17.75 mg/g, respectively. Percentage dye removal for 25 and 50 mg/l dye and 0.2 g/100 cm^3 adsorbent was 87.64 and 71 %, respectively.

Removal efficiency increases with increasing amount of adsorbent, and the initial concentration of dye provides the driving force which overcomes resistance to mass transfer of RR198 dye between the aqueous and solid phases [26]. These results agree with those reported by Elkady et al. [2], who found that removal of Remozal Red 198 by eggshell bio-composite beads increases up to a specific limit and then remains almost constant. They reported that increasing adsorption with increasing amount of adsorbent can be attributed to increased adsorbent surface area and the availability of more adsorption sites [2]. Mittal et al. [27] considered the potential use of coconut husk for removal of quinoline yellow dye from wastewater. Their results showed that adsorption increases with increasing amount of adsorbent.

Effect of initial dye concentration and contact time

In this study, for detection of the effect of dye concentration and contact time on adsorption of RR198, the amount of adsorbent was 0.2 g/100 ml at pH 3 (Fig. 4). The results showed that as the initial concentration of the dye was increased, percentage removal decreased, although the actual amount of dye adsorbed per unit mass of pomegranate seed powder increased with increasing of initial dye concentration, because the initial concentration is an important driving force overcoming resistance to mass transfer of the dye between the aqueous and solid phases. Therefore, an increase in the initial concentration of the dye enhances adsorption of the dye [12, 26]. This increase is also because of reduced resistance to uptake of the dye from solution [12]. These results accord well with those of other investigators, for example Kamal Amin [12], who studied removal of direct blue-

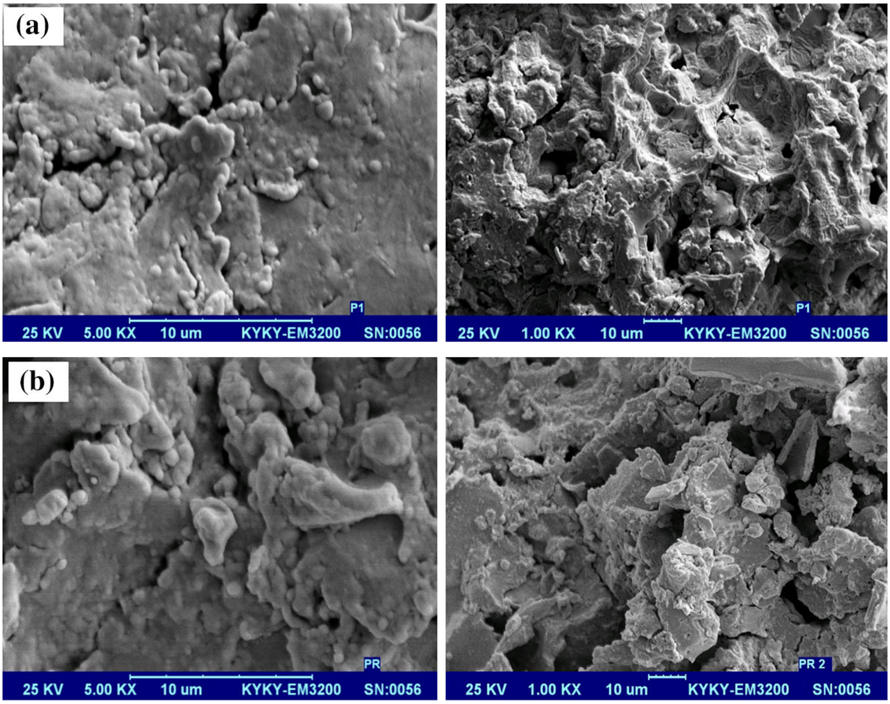

Fig. 2 Scanning electron microscope (SEM) images of pomegranate seed powder at different magnification: **a** before dye sorption and **b** with dye adsorbed

106 dye from aqueous solution on new types of activated carbon developed from pomegranate peel. Hameed et al. [28] studied removal of cationic dye from aqueous solutions by use of pineapple stem waste and showed that the amount of dye adsorbed (q_e) increased when dye concentration increased and remained constant after equilibration.

The contact time between adsorbate and adsorbent is the most important design condition affecting the performance of the adsorption process. Investigation of the results showed that increasing the contact time to 30 min resulted in an increase in the adsorption capacity (q_e) for RR198 dye which then became constant. The results showed that the amount of dye adsorbed decreased with increasing time; this is because of decreasing dye concentration and a decrease in the number of active sites on the surface of the adsorbent. The adsorption equilibrium time for removal of RR198 dye by pomegranate seed powder was up to 30 min. The efficiency of removal of the dye at 30 min for 25 and 50 mg/l initial concentrations was 79.44 and 62.6 %, respectively. The amount of dye adsorbed per mass unit of adsorbent (q_e) for concentrations of 25 and 50 mg/l at equilibrium (30 min) was 9.93 mg/g and 15.65 mg/g, respectively. Similar results were obtained by Mittal et al. [27], who showed that adsorption of quinoline yellow dye by coconut-husk reached equilibrium after 30 min and then remained constant.

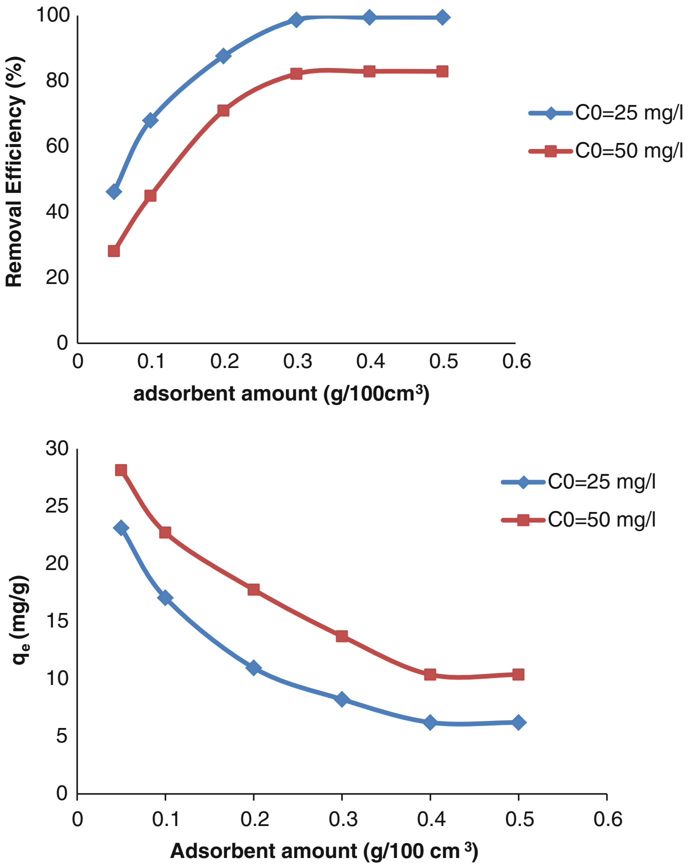

Fig. 3 Effect of adsorbent amounts on removal efficiency and adsorption capacity for RR198 dye

Adsorption isotherms

To determine the adsorption capacity of pomegranate seed powder, the experimental data points were fitted to the Langmuir and Freundlich isotherm equations. The constants of the isotherm equations were calculated and are listed in Table 2. Values of the correlation coefficient (R^2) are regarded as a measure of the goodness-of-fit of experimental data to the isotherm model. The R^2 values for the Freundlich isotherm were higher than 0.9 ($R^2 = 0.948$ and 0.936 for dye concentrations of 25 and 50 mg/l, respectively), indicative of a good fit with this isotherm model (Fig. 5). According to our results, $1/n$ values were <1 ($1/n = 0.256$ and 0.625 for dye concentrations of 25 and 50 mg/l, respectively), which confirms adsorption of the dye by this biosorbent under the conditions of this research is favorable and suggests that the process was controlled by chemisorption.

According to the Freundlich isotherm, adsorption of RR198 is single-layer adsorption on a homogeneous adsorbent surface. Kamal Amin [12] also studied the removal of direct blue-106 dye on new types of activated carbon developed from

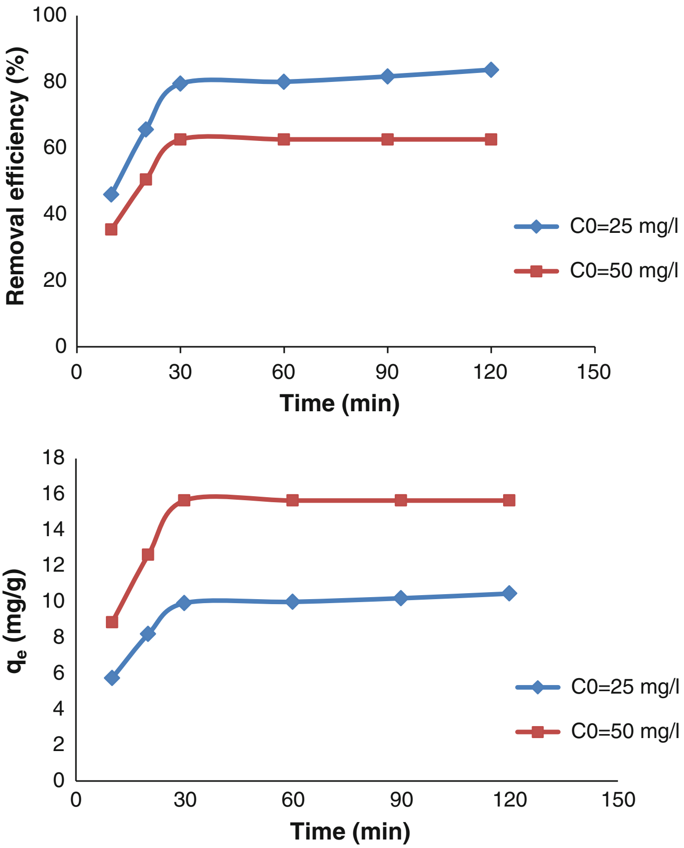

Fig. 4 Effect of contact time on removal efficiency and adsorption capacity for RR198 dye

Table 2 Characteristics of adsorption isotherms

Initial concentration of dye (mg/l)	Langmuir isotherm		Freundlich isotherm	
25	B (l/mg)	0.203	K (mg/g) (l/mg)$^{1/n}$	10.21
	$q_{\max}$ (mg/g)	76.92	$1/n$	0.256
	R^2	0.896	R^2	0.948
	R_L	0.165		
50	B (l/mg)	33.27	K (mg/g) (l/mg)$^{1/n}$	3.01
	$q_{\max}$ (mg/g)	1.67	$1/n$	0.625
	R^2	0.914	R^2	0.936
	R_L	6×10^{-4}		

pomegranate peel. His results showed that the Langmuir, Freundlich, and Temkin isotherms were in good concordance with the data ($R^2 > 0.9$), with agreement being best for the Freundlich model [12]. Yalçın et al. [29] studied removal of basic

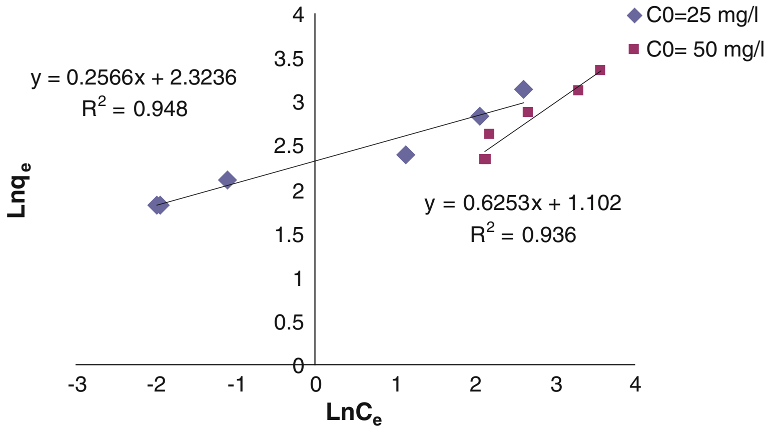

Fig. 5 Freundlich isotherm plots for adsorption of RR198 dye

yellow 2, basic green 4, and reactive orange 113 by use of raw and treated *Phanerochaete chrysosporium*. In their study, the Freundlich isotherm was a good fit with the equilibrium data [29].

Adsorption kinetics

Adsorption kinetics are used to explain the mechanism and characteristics of adsorption [30]. In this study, pseudo-first-order and pseudo-second-order kinetic models were analyzed. Data for each model were calculated and are listed in Table 3. For the pseudo-first-order reaction model the theoretical value of q_e ($q_{e,cal}$) is lower than the experimental value ($q_{e,exp}$), indicating the adsorption process does not follow this model. The R^2 values for the pseudo-second-order equation for both initial dye concentrations were closer to unity. The values of the correlation coefficient (R^2) for concentrations 25 and 50 mg/l in the pseudo-second-order model were 0.998 and 0.996, respectively (Fig. 6). Thus, the pseudo second-order model provides good correlation for adsorption of RR198 on pomegranate seed powder, showing that adsorption of RR198 by pomegranate seed powder is most likely to be controlled by a chemisorption process. In the study by Kamal Amin [12] on removal of direct blue-106 dye by use of new types of activated carbon developed from pomegranate peel, the adsorption kinetics followed a pseudo-second-order kinetic model, with good correlation ($R^2 > 0.99$). Similar results were also reported by Wang et al. [31] and Yu et al. [32].

Conclusion

Increasing the abount of adsorbent and contact time led to an increase in the rate of adsorption, and that increasing the initial concentration dye led to decrease of removal efficiency. Isotherm studies showed that removal of RR198 by use of

Table 3 Kinetic constants for adsorption of RR198 by pomegranate seed powder

Initial concentration of dye (mg/l)	Pseudo-first-order		Pseudo-second-order	
25	$q_{e(calc.)}$ (mg/g)	1.84	q_e(mg/g)	11.11
	$q_{e(exp.)}$ (mg/g)	10.955	K_2	0.013
	K_1 (min^{-1})	0.017	R^2	0.998
	R^2	0.763		
50	q_e(calc.) (mg/g)	1.383	q_e(mg/g)	16.67
	q_e(exp.) (mg/g)	17.75	K_2	0.011
	K_1 (min^{-1})	0.010	R^2	0.996
	R^2	0.502		

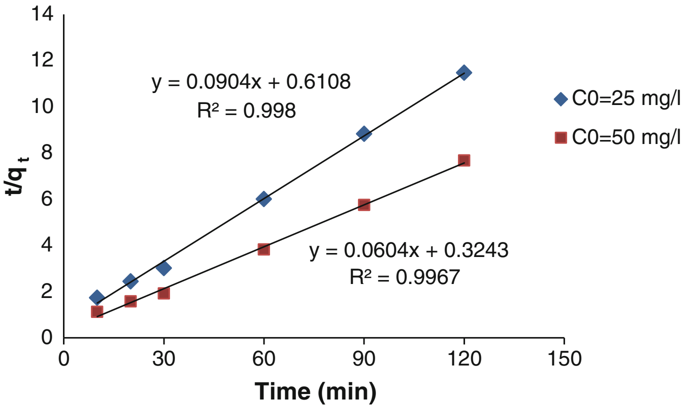

Fig. 6 Pseudo-second-order kinetics plots for adsorption of RR198 dye

pomegranate seed powder most closely followed the Freundlich isotherm. Study of the adsorption kinetics showed that adsorption of RR198 followed the pseudo-second-order equation. Because of the high cost of synthetic adsorbents, development and application of inexpensive and natural adsorbents is essential. The results of this study revealed that pomegranate seed waste can be used as an effective adsorbent for removal of RR198 dye from aqueous solutions.

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