

Adsorption of anionic azo dye from aqueous solution using *Strychnos potatorum* Linn seeds: isotherm and kinetic studies

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Abstract Dyes from several industry effluents are major concern of environmental pollution. In this study, the efficiency of a cost-effective natural adsorbent *Strychnos potatorum* Linn (Fam: Loganiaceae) seeds on removing reactive orange-M2R dye from aqueous solution was investigated using batch adsorption isotherms at 30 ± 1 °C and 120 rpm. The efficacy of the adsorbent system was also studied at various parameters viz. contact time, pH, adsorbent dosage and initial dye concentration. Maximum adsorption ($q_e = 9$ mg/g) was observed at pH 2.0, equilibrium time 6 h, initial dye concentration of 100 mg/L and adsorbent dosage of 0.2 g/100 mL. The unit adsorption of dye, q_e (mg/g) increased with increase in contact time and initial dye concentration, while it decreased with increase in adsorbent dosage. The adsorption kinetic studies revealed that it follows pseudo-second-order reaction model. Equilibrium adsorption data followed both Langmuir and Freundlich isotherms with good agreement. These investigations suggest that *Strychnos potatorum* Linn seeds (SPS) could be used as a low-cost adsorbent in treating textile effluents for dye removal applications. The adsorbent (SPS) was also characterized using Fourier transform infrared spectroscopy, powder X-ray diffraction, and scanning electron microscopic technique.

Keywords *Strychnos potatorum* Linn seeds · Adsorption · Environment · Dye removal · Reactive orange-M2R

Introduction

Many researchers have tried to provide methods for pollution prevention by providing adequate treatment of the effluents from industrial and residential sources (Sid Kalal et al. 2012). One of the major problems of water bodies (rivers and lakes) is the release of waste water from several dyeing industries (Khan et al. 2004). In India, many industries viz, textile, leather, printing, food etc., use dye as a coloring agent. Around 10,000 commercial dyes are available in the market and among these, 60–70 % are azo dyes. In general, azo dyes are categorized based on its chemical structure (monoazo, diazo, triazo and tetraazo dyes) and other by its application (acidic, basic, direct, reactive, pigments and disperse). In textile industry, for dyeing purpose, 30–60 L of water is consumed per kg of cloth dyed. It is reported that after processing, 10–15 % of the dyes present in the effluent contaminates local water bodies (Sivaraj et al. 2000). The effluent imparts color and reduces the sunlight penetration and thereby retards the growth of flora and fauna by affecting photosynthesis. The effluent also contains various carcinogens and mutagens which are detrimental to the mankind (Gregory et al. 1991). Ozacar and Sengil (2003) reported that several harmful chemicals in the effluent cause chelation of metal ions. This in turn, depletes the dissolved O₂ concentration which adversely affects fish and other aquatic organisms by producing micro-toxicity.

Most of the dyes used in dyeing section are stable against biological and photo-degradation. Presently, several chemical and physical treatment methods are available to treat the dye industry effluents. But, these methods are not economical and effective against wide range of dyes. Several works have been reported on conventional and non-conventional adsorbents for the dye removal from

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aqueous solution (Pollard et al. 1992; Namasivayam et al. 2001; Ramakrishna and Viraraghavan 1997). The liquid phase adsorption was found to be highly efficient for the removal of color (Garg et al. 2004a, b). In addition, regeneration of adsorbent also should be taken into account to make the method an economically viable one.

In this present study, the use of *Strychnos potatorum* Linn Seeds (SPS) as an adsorbent for the removal of reactive orange-M2R (RO-M2R), a monoazo dye was explored. Kirtikar and Basu (1933) reported that *Strychnos potatorum* Linn (deciduous tree) is widely available in central and southern parts of India, Sri Lanka and Burma. The seeds of this tree are known for purifying mud water and thus have its common name as “clearing nut tree”. Besides, there are several reports available on medicinal importance of the fruits of this tree (Kirtikar and Basu 2000). The aim of the present work is to find out the ability of SPS as a low-cost, naturally available adsorbent for the removal of RO-M2R dye under optimized conditions.

This research work was carried out at Department of Biotechnology, Bannari Amman Institute of Technology, Tamilnadu, India during May to October 2013.

Materials and methods

Adsorbent and adsorbate

Strychnos potatorum Linn seeds was purchased from the Parrys market, Chennai, India. The SPS was dried in hot air oven at 70 °C for 48 h and powdered using a grinder. The powdered adsorbent was sieved through a 250 µm diameter mesh. RO-M2R was procured from local textile dyeing unit, Tirupur, Tamil Nadu, India. Other chemicals used in the experiment were of highest purity and of analytical grade. The absorbance of samples was measured using UV–Vis Spectrophotometer (Perkin Elmer, Lambda-35).

Batch adsorption studies

The batch dye adsorption studies were conducted by agitating 100 mL volume of reactive dye solution of known concentration (5–200 mg/L) with 0.2 g of SPS in 250 mL conical flask. Dye solution was prepared by adding distilled water in order to minimize possible interferences caused by tap water. Agitation was carried out in a mechanical shaker (Biotech Scigenics Orbitek, India) at desired temperature of 30 ± 1 °C and allowed it to shake for 1–48 h to attain equilibrium conditions. The samples were withdrawn at different time intervals to determine the change in absorbance. After completion of experiment, the collected samples were centrifuged (REMI Instruments Ltd, India) at

10,000 rpm for 10 min to separate the adsorbent from the dye solution. The absorbance was spectrophotometrically analyzed in the visible region (~490 nm). The experiments were carried out in triplicates.

The amount of dye adsorbed at equilibrium ‘ q_e ’ was calculated by the following equation,

$$q_e = \left(\frac{C_0 - C_e}{W} \right) \times V \quad (1)$$

where C_0 and C_e (mg/L) are the initial and equilibrium concentrations of dye solution, respectively. V is the total volume of the solution (L) and W is the weight (g) of SPS used for the study.

The percentage removal of dye from aqueous solution was calculated using the following equation:

$$\% \text{ dye removal} = \left(\frac{C_0 - C_e}{C_0} \right) \times 100 \quad (2)$$

Effect of pH on adsorption

The effect of pH on the removal of RO-M2R dye by the adsorbent was studied at different pH (2–10). The pH of the solution was adjusted using small amounts of 0.1 N NaOH or 0.1 N HCl. The experiments were carried out by agitating 100 mL of 100 mg/L initial dye concentration with a constant adsorbent mass of 0.2 g/100 mL for an equilibrium time of 6 h at 120 rpm and 30 ± 1 °C. The conical flask containing the dye solution without the adsorbent was maintained as control.

Effect of contact time on adsorption

The contact time influence in the range of 1–48 h was investigated with different initial dye concentrations (20, 50, 100, 150, 200 mg/L) at temperature 30 ± 1 °C with constant adsorbent dosage of 0.2 g/100 mL.

Effect of initial adsorbent dosage and initial dye concentration on adsorption

The effect of initial adsorbent dosage on dye removal was investigated by changing the initial adsorbent dosage between 0.05 and 1.0 g/100 mL, while other experimental conditions such as initial dye concentration (100 mg/L), temperature (30 ± 1 °C) and pH 2 were maintained for an equilibrium time of 6 h. For the effect of initial dye concentration on the percentage removal of RO-M2R by SPS, the study was conducted at the experimental conditions of fixed adsorbent mass of 0.2 g/100 mL with different initial dye concentrations (5–200 mg/L), pH 2 and temperature (30 ± 1 °C) kept for an equilibrium time of 6 h.

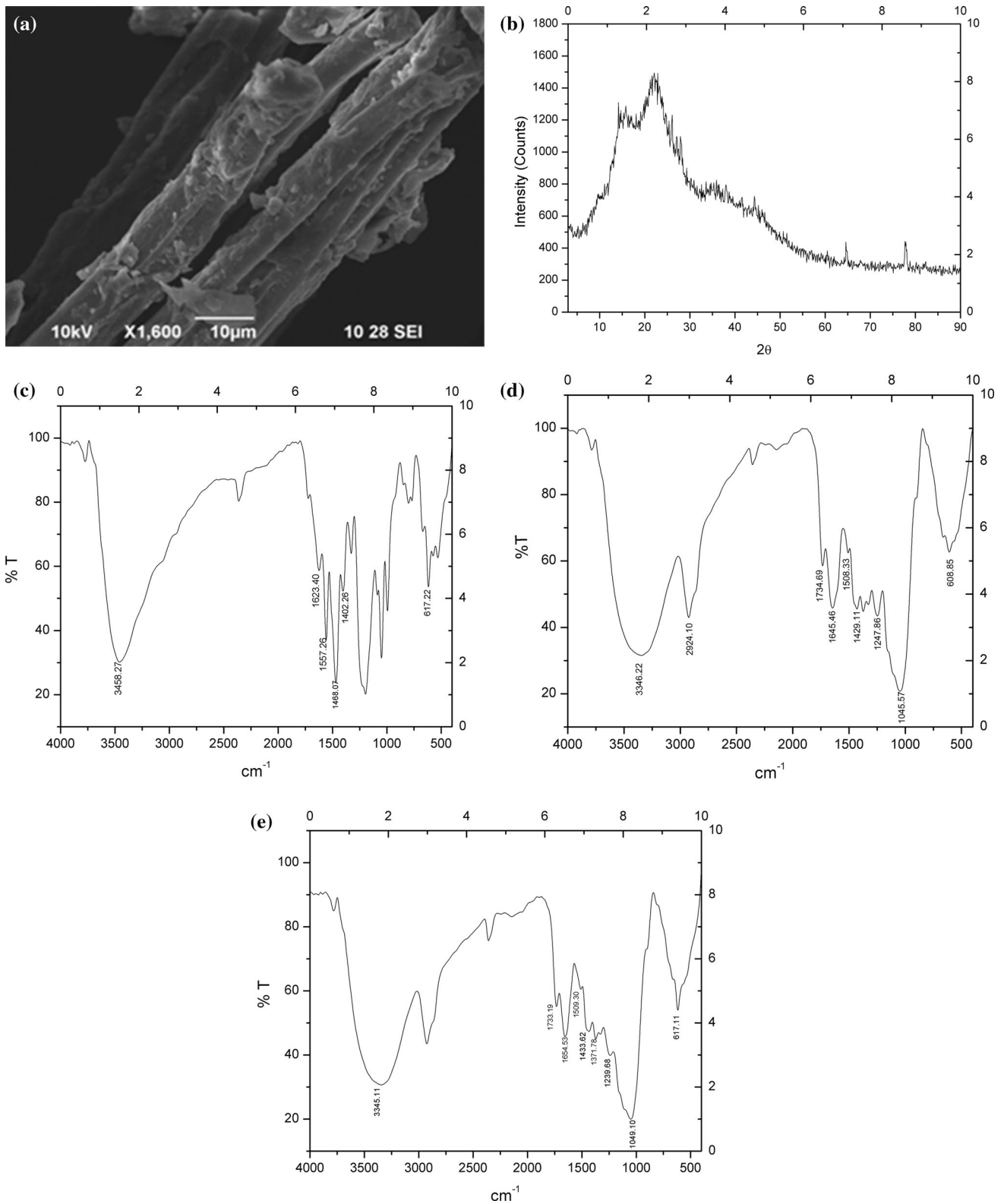


Fig. 1 Characterization of adsorbent. **a** SEM micrograph of SPS. **b** XRD pattern of SPS. **c** FTIR spectrum of RO-M2R. **d** FTIR spectrum of SPS. **e** FTIR spectrum: after adsorption of RO-M2R by SPS

Adsorption kinetics

The adsorption kinetic study of RO-M2R dye on SPS was conducted with an initial dye concentration of 100 mg/L and 0.2 g of adsorbent material. The experiment was conducted at pH 2 and temperature 30 ± 1 °C for an equilibrium time of 6 h. At various interval of time, samples were collected and centrifuged at 10,000 rpm for 10 min and absorbance was measured spectrophotometrically. The amount of adsorption at time t , q_t (mg/g) was calculated by the following equation:

$$q_t = \frac{(C_0 - C_t)V}{W} \quad (3)$$

where C_0 and C_t (mg/L) are the concentrations of dye at initial and at time t , respectively, V is the volume of the solution (L) and W is the weight of adsorbent used in (g).

SEM, XRD and FTIR analysis

In this study, scanning electron microscopy (SEM), powder X-ray diffraction (XRD) and Fourier transform infrared (FTIR) spectrophotometry analyses were performed in order to characterize the adsorbent SPS. The SEM used is JEOL 6390, Japan with a magnification of 1,600 \times , XRD-6000 Shimadzu, Japan and FTIR, ABB MB3000 with spectra recorded in the range of 400–4,000 cm^{-1} .

Results and discussion

Characterization of adsorbent

Scanning electron microscopy analysis of SPS was carried out in order to study the morphological and surface characteristics of the adsorbent. Figure 1a shows the SEM micrograph of the adsorbent showing bundle-like structures with a heterogeneously textured surface. This surface consistency may aid the adsorption of the dye molecule over the SPS surface.

The X-ray diffraction analysis of SPS illustrated a highly crystalline nature indicated by the presence of broad main peak centered at 2θ of 22° , a characteristic peak of cellulosic material (Fig. 1b). The presence of the main peak ascertains the presence of highly organized crystalline cellulose content of SPS. The crystalline nature of the adsorbent may assist the dye adsorption over the material surface.

The FTIR is an effective tool to investigate various functional groups present in the prepared samples. In the case of pure RO-M2R, the obtained peaks around 3,458 and 1,623 cm^{-1} belong to OH stretching and aromatic C–C stretching, respectively. The peaks around 1,557 and

1,468 cm^{-1} resemble C–C double-bond stretching vibrations and for alkyl acetals (C–H stretching) at 1,402 cm^{-1} . The functional group C–N stretching due to amines displayed at 1,196 cm^{-1} . The peaks arrived at 617 cm^{-1} depicts C–Cl stretching (Fig. 1c). The FTIR spectra of before and after adsorption of RO-M2R by SPS are shown in Fig. 1d, e. By comparing both the spectra, the shifting of peaks to higher wave number and the presence of amine group in the FTIR spectrum of after adsorption process confirms the presence of RO-M2R dye on the surface of the sample. All the observations are in good agreement with the literature (Jagwani et al. 2013).

Effect of pH and contact time on dye removal

pH is an important parameter for an adsorption experiment which directly has an impact on adsorption capacity of the adsorbent. The amount of dye adsorbed by adsorbent (mg/g) decreased, when initial pH was increased from 2 to 10 (Fig. 2a). When the pH increased from 2 to 6, the amount of dye adsorbed decreased from 9 to 3 mg/g. With further increase in pH to 10, there was no significant change in the adsorption of dye. A maximum adsorption of 9.36 mg/g was observed at pH 2. Similar adsorption behavior with respect to variation in pH was reported in the studies of Newcombe and Drikas (1997). High adsorption of anionic dye RO-M2R at low pH indicates that the system seems to be “below the point of zero charge”. The studies of Giles et al. (1974) suggest that the overall surface of adsorbent becomes positively charged and the greater degree of adsorption observed may be due to hydrophobic interactions. The percentage of dye removal increased with increase in contact time (Fig. 2b). The adsorption percentage increased drastically at initial hours and it slowly attained an equilibrium value. The maximum percentage removal of RO-M2R dye was attained within 6 h and after that there was no significant increase in dye removal and it attained an equilibrium value.

Effect of initial adsorbent dosage and initial dye concentration

The results showed that the percentage removal of RO-M2R dye increased from 18.05 to 51.81 % with increase in initial adsorbent dosage from 0.05 to 1.0 g/100 mL (Fig. 3a). The maximum dye removal of 51.81 % was observed at an adsorbent dosage of 1.0 g/100 mL. Garg et al. (2004a, b) reported that the increase in adsorbent dosage results in increased surface area and thereby increases the availability of adsorption sites for binding. While percentage adsorption increased, the unit adsorption (q_e) decreased from 36.11 to 5.18 mg/g with an increase in adsorption mass. Namasivayam et al. (1998) reported that

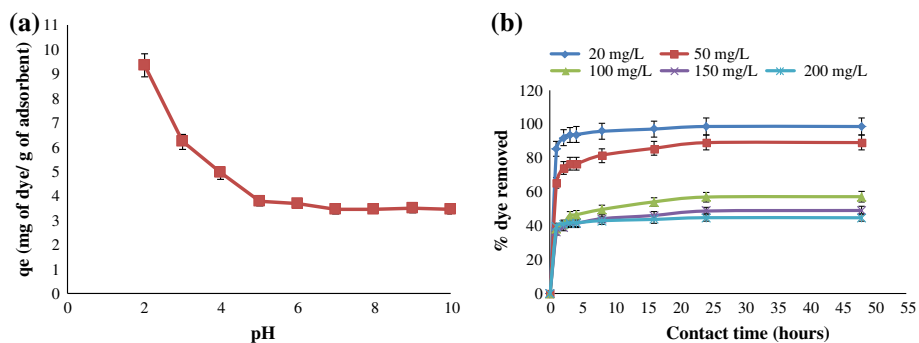


Fig. 2 a Influence of pH on RO-M2R dye removal onto adsorbent (SPS) from aqueous solution (temperature: 30 ± 1 °C, agitation speed: 120 rpm, pH: 2–10, contact time: 6 h), **b** Influence of contact

time on RO-M2R dye removal onto adsorbent from aqueous solution (temperature: 30 ± 1 °C, agitation speed: 120 rpm, pH: 2)

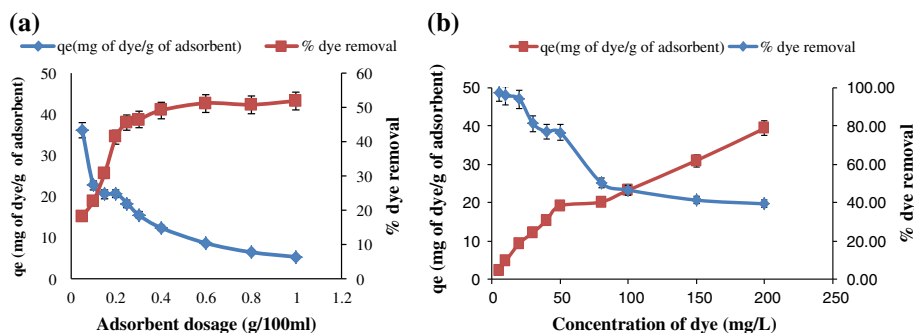


Fig. 3 a Influence of initial adsorbent dosage on RO-M2R dye removal onto adsorbent (SPS) from aqueous solution (temperature: 30 ± 1 °C, agitation speed: 120 rpm, pH: 2), **b** Influence of initial

dye concentration on RO-M2R dye removal onto adsorbent from aqueous solution (temperature: 30 ± 1 °C, agitation speed: 120 rpm, pH: 2)

this effect may be due to overlapping or add up of adsorbent binding sites available to RO-M2R and it increases the diffusion path length of dye to reach the adsorption binding site.

This study revealed that the percentage removal of RO-M2R decreased from 97.62 to 39.41 % with increase in the initial concentration of dye (Fig. 3b). While the percentage removal dye decreased, the unit adsorption (q_e) increased from 2.44 to 39.41 mg/g with increase in the initial dye concentration. This may be attributed to the presence of higher residual dye concentration for higher initial dye concentration. It is evident from the studies of Ju et al. (2006) that the adsorption capacity for the RO-M2R increases with initial dye concentration because of the concentration gradient. It was also observed that the active sites on the adsorbent for dye removal may become saturated as the dye concentration increases.

Adsorption isotherms

The adsorption isotherm is of basic importance in the modeling of adsorption. Bello et al. (2010) reported that the adsorption isotherm expresses the distribution of dye

between the liquid phase and the adsorbent phase, when the process reaches its equilibrium state. In order to quantify the adsorption affinity of SPS for RO-M2R and to find out the best model for the design of adsorption process, the adsorption equilibrium data should exactly fit into different isotherm models (Langmuir and Freundlich isotherm models).

The Freundlich isotherm assumes the multilayer adsorption and for the adsorption on heterogeneous surfaces and it is represented by an equation below,

$$\log q_e = \log K_f + \frac{1}{n} \log C_e \tag{4}$$

where q_e is the amount of dye adsorbed per unit mass of adsorbent (mg/g), C_e is concentration of dye at equilibrium (mg/L), K_f roughly shows the adsorption capacity and $\frac{1}{n}$ represents the adsorption intensity. The coefficients K_f and n can be estimated from intercept and slope in the graph of $\log q_e$ vs $\log C_e$ (Fig. 4a) and presented in Table 1. The linear plot for the graph of 20, 50, 100, 150, 200 mg/L dye concentrations substantiates the applicability of the Freundlich isotherm model ($R^2 > 0.97$). The n value greater than 1 represents favorable adsorption condition. The

Fig. 4 Adsorption isotherms. **a** Freundlich plot. **b** Langmuir plot

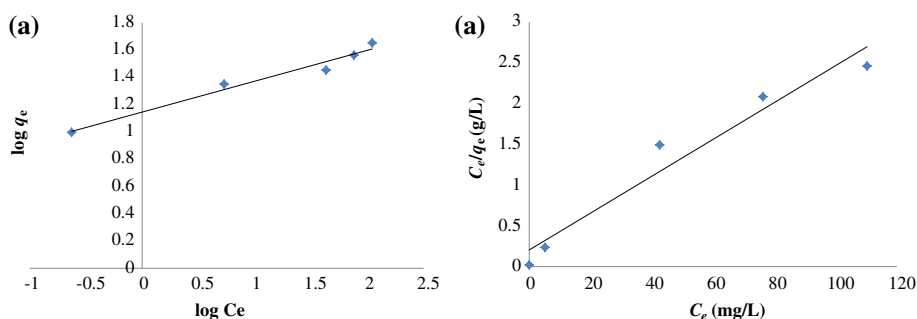


Table 1 Isotherms Constants

Langmuir coefficients			Freundlich coefficients		
K_L (L/mg)	S_m (mg/g)	R^2	K_f (mg/g)	n	R^2
0.114	44.247	0.951	14.01	4.405	0.972

results suggest that the SPS favorably adsorbs RO-M2R dye.

Weber and Chakravorti (1974) indicated that the energy term in the Langmuir equation varies as a function of the surface exposure. The adsorption of RO-M2R in the current study was analyzed using Langmuir adsorption model which is represented by the following equation (Inbaraj et al. 2002; Annadurai et al. 1999; Stephen et al. 1989; El-Geundi 1991; Lata et al. 2007),

$$\frac{C_e}{q_e} = \frac{1}{K_L S_m} + \frac{C_e}{S_m} \quad (5)$$

where S_m is the maximum amount of adsorption per unit mass of adsorbent (mg/g) resembles the complete monolayer surface coverage (Pavan et al. 2008), C_e represents the concentration of dye at equilibrium (mg/L), K_L represents the Langmuir constant (L/mg). Fytianos et al. (2000) reported that the Langmuir isotherm assumes uniform strategies of adsorption. S_m helps in the comparison of adsorption capacity. The coefficients K_L and S_m can be calculated from intercept and slope in the graph of $\log C_e/q_e$ vs $\log C_e$ (Fig. 4b) and is presented in Table 1. The linear plot for the graph of 20, 50, 100, 150 and 200 mg/L dye concentrations substantiates the applicability of the Langmuir isotherm model ($R^2 > 0.95$). The essential uniqueness of Langmuir isotherm can be expressed by a dimensionless constant separation factor for equilibrium parameter, R_L (Hall et al. 1966) which can be calculated as below,

$$R_L = \frac{1}{(1 + K_L C_0)} \quad (6)$$

where C_0 is the initial concentration of dye (mg/L) and K_L is the Langmuir constant (L/mg). The values of R_L indicate the shape of the isotherm. The shape of the isotherm may

be interpreted, when $R_L > 1$ as unfavorable, $R_L = 1$ as linear, $1 > R_L > 0$ as favorable and $R_L = 0$ as irreversible adsorption. The magnitudes of R_L for the adsorption process studied at different initial dye concentrations ranged between 0 and 1, thus indicating a favorable adsorption process between SPS and RO-M2R dye.

Adsorption kinetic studies

To determine the potential rate-controlling steps involved in the mechanism of solute sorption onto the adsorbent, kinetic studies were carried out. Several kinetic models can be used to investigate the adsorption process. In this study, pseudo-first-order, pseudo-second-order and intra-particle diffusion models were employed to understand the dependency of time over the concentration distribution of the solute both in bulk solution and solid adsorbent phases.

The Lagergren's pseudo-first-order equation is generally expressed as (Yuh-Shan Ho 2004; Lata et al. 2008),

$$\log(q_e - q_t) = \log(q_e) - \left(\frac{k_1}{2.303}\right)t \quad (7)$$

where q_e and q_t (mg/g) are the amount of dye adsorbed per unit mass of adsorbent at equilibrium and at time t (min), respectively, k_1 is the rate constant of pseudo-first-order kinetics (1/min). The linear plot of $\log(q_e - q_t)$ as a function of adsorption time (t) is shown in Fig. 5a. The values of k_1 and q_e can be calculated from the slope and intercept of the plot, respectively, are shown in Table 2.

Kinetic data of the present study were employed in the pseudo-second-order rate expression (Poots et al. 1978),

$$\frac{t}{q_t} = \left(\frac{1}{k_2 q_e^2}\right) + \left(\frac{t}{q_e}\right) \quad (8)$$

where k_2 is the rate constant of pseudo-second-order kinetics (g/mg/min). The linear plot of (t/q_t) as function of time (t) is shown in Fig. 5b. The values of q_e and k_2 can be calculated from the slope and intercept of the plot, respectively, are shown in Table 2.

Singh and Rawat (1994) reported that the adsorption process includes the transport of adsorbate molecules from

Fig. 5 Adsorption Kinetics. **a** Lagergren’s first-order plot. **b** Pseudo-second-order plot. **c** Intra-particle diffusion model for adsorption

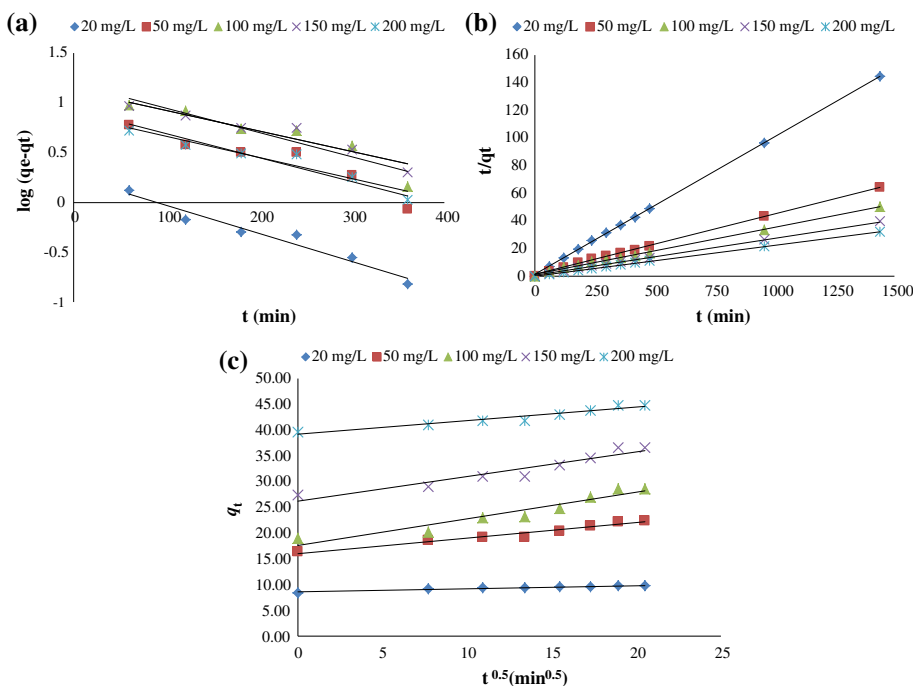


Table 2 Linearized kinetic coefficients for the dye sorption onto SPS

Dye (mg/L)	Theoretical (q_e) (mg/g)	Pseudo-first order			Pseudo-second order			Intra-particle diffusion		
		$(q_e)_{cal}$	K_1	R^2	$(q_e)_{cal}$	K_1	R^2	K_i	C	R^2
20	9.88	1.759	0.384	0.9483	9.921	0.017	1.0000	0.032	8.909	0.8049
50	22.31	8.602	0.337	0.8867	22.573	0.002	0.9996	0.157	17.280	0.9039
100	28.51	15.646	0.341	0.8752	29.070	0.001	0.9992	0.274	19.598	0.9088
150	36.57	13.530	0.288	0.9228	37.037	0.001	0.9995	0.264	27.849	0.9321
200	44.82	7.434	0.296	0.9205	45.045	0.003	0.9999	0.145	40.080	0.9302

bulk solution to the interior surface of the pores. The applicability of intra-particle diffusion was investigated using intra-particle diffusion model (Ozcan and Ozcan 2005; Weber and Morris 1963).

$$q_t = k_i t^{0.5} + C \tag{9}$$

where q_t is the unit adsorption of dye (mg/g) at time, t (min), k_i is the intra-particle diffusion rate constant (mg/g.min^{0.5}) and C represents boundary layer effect which indicates that, greater the value of C larger the boundary layer thickness (Lee et al. 1999). The linear plot of q_t versus $t^{0.5}$ of Eq. 9 is shown in Fig. 5c. The values of k_i and C can be calculated from the slope and intercept of the plot, respectively, are shown in Table 2.

Conclusion

In this study, the maximum dye removal was observed at the experimental conditions of pH 2, equilibrium time

6 h, temperature 30 ± 1 °C and an adsorbent dose of 0.2 g/100 mL. The adsorption capacity was found to change with adsorbent dosage, initial dye concentration and pH. Adsorption kinetic studies showed that the rate of adsorption obeyed pseudo-second-order kinetics model with good correlation. Equilibrium data followed both Langmuir and Freundlich isotherm models well (Lee et al. 1999; Ong et al. 2007; Malik 2003). The fitness of data with Langmuir model confirms the monolayer coverage of the adsorbate (RO-M2R) onto the outer surface of the adsorbent (SPS). SPS is economically cheap, and hence, regeneration is not necessary. *Strychnos potatorum* Linn trees are widely available in India, and its seeds are available at zero cost or negligible price. The data provided in the study may be useful in designing and fabricating a economically feasible treatment process using batch or continuous stirred tank reactors for the removal of RO-M2R from industrial effluents by adsorption on SPS.

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