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Adsorptive removal of Erythrosine dye onto activated low cost de-oiled mustard

Rajeev Jain*, Shalini Sikarwar

Department of Environmental Chemistry, Jiwaji University, Gwalior 474011, India

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ABSTRACT

The present paper is aimed to investigate and develop cheap adsorption methods for colour removal from wastewater using waste material de-oiled mustard as adsorbent. De-oiled mustard, a biosorbent, was successfully utilized for removing a water-soluble xanthene dye, Erythrosine from wastewater. Kinetic studies of adsorption of Erythrosine at de-oiled mustard were carried out at 30 °C, using aqueous solutions with 5×10^{-5} M concentration of Erythrosine. The adsorption process followed a pseudo-first order model. The equilibrium process can be well described by both Freundlich and Langmuir models, at 30, 40 and 50 °C. Free energy of adsorption (ΔG°), enthalpy (ΔH°), and entropy (ΔS°) changes were calculated to predict the nature of adsorption. The estimated values for ΔG° were -12.81×10^3 and -12.57×10^3 over activated carbon and activated de-oiled mustard at 203 K (30 °C), indicate toward a spontaneous process. The positive value for ΔH° indicates that the adsorption of Erythrosine dye to de-oiled mustard is an endothermic process.

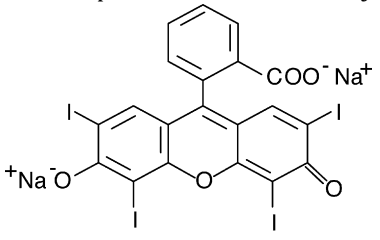
1. Introduction

Textile wastewater is generally high in both colour and organic content. Effluents discharged from dyeing industries are highly coloured and they can be toxic to aquatic life in receiving waters [1,2]. Colour removal from textile effluents has been given much attention in the last few years, not only because of its potential toxicity, but also mainly due to its visibility problems [3,4]. The total dye consumption of the textile industry worldwide is in excess of 107 kg/year, and an estimated 90% of this ends up on fabrics. Consequently, 1000 tones/year or more of dyes are discharged into waste streams by the textile industry only worldwide [5].

Among several chemical and physical methods, the adsorption on to activated carbon has been found to be superior to other techniques in water-re-use methodology because of its capability for adsorbing a broad range of different types of adsorbates efficiently, and simplicity of design. Increasingly stringent legislation on the decontamination of wastewater has created interest for the use of waste activated carbon for this purpose [6–10]. However, commercially available activated carbons are still considered expensive [11] and therefore, their use may imply carrying out regeneration and reactivation procedures [12]. Thus, many researchers researched for cheaper substitutes, which are relatively inexpensive, and are at the same time endowed with reasonable adsorptive capacity [13–28]. These studies include the use of coal [29], fly ash [30], activated clay [31], palm-fruit bunch [32], bagasse pith [33], cellulose based waste [34], peat, bentonite, slag and fly ash [35], activated sludge [36], rice husk and gram husk [37–39], etc. The object of the present investigations has been to evaluate the efficiency of removal of Erythrosine using activated carbon (AC) and activated de-oiled mustard (ADM). Activated de-oiled mustard are oil mill waste material, which is easily available, cheap, and economically advantageous. The effect of adsorbent dose, pH, particle size, temperature, initial dye concentration, and equilibrium time has been studied and obtained results are discussed.

2. Materials and methods

The dye Erythrosine (A) was obtained from M/s Merck and its 0.01 M stock solution was prepared in double distilled water. To prepare various solutions at desired concentrations from the stock solution, double distilled water was used for necessary dilutions. All reagents used in the present work were of analytical grade.



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* Corresponding author. Tel.: +91 7512442766; fax: +91 7512346209.
E-mail address: rajeevjain54@yahoo.co.in (R. Jain).

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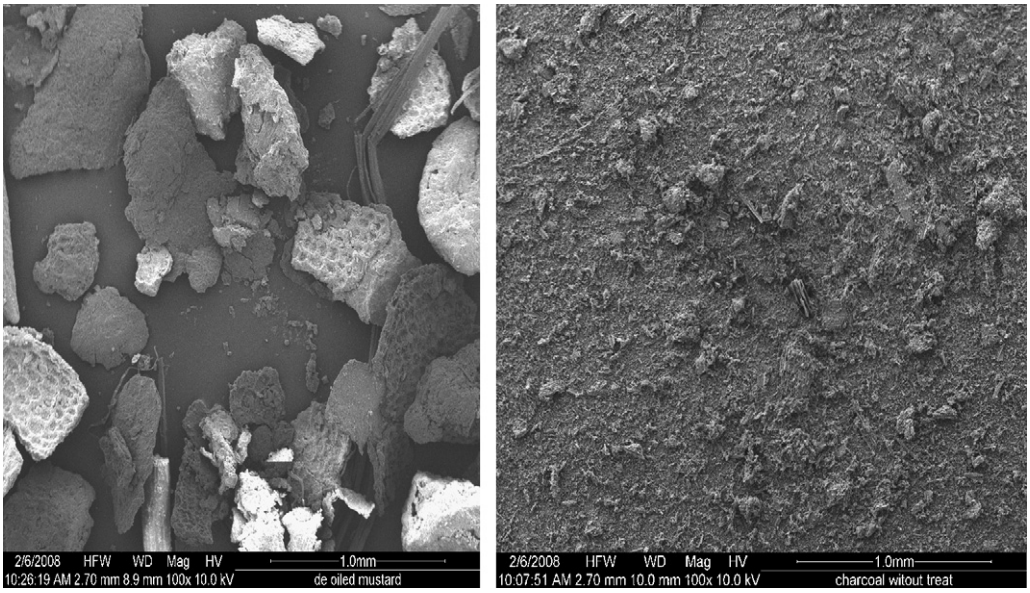

Fig. 1. SEM micrographs of (a) AC and (b) ADM adsorbents.

Adsorbent activated carbon (AC) was purchased from M/s Merck and used as received, while de-oiled mustard was collected from the local oil mill industries. The dose of adsorbent was varied from 0.06 to 0.72 g/l for activated carbon (AC) and from 2.5 to 20 g/l for activated de-oiled mustard (ADM). All pH measurements were carried out with a decibel DB 1011 digital pH meter, fitted with a glass electrode and COD digestion apparatus (Spectra-Lab-2015 S) was used for determining COD of the solutions. Absorbance measurements were recorded on a spectronic 20D+ thermospectronic spectrophotometer over the wavelength range 200–800 nm.

2.1. Adsorbent development

De-oiled mustard was cleaned, thoroughly washed with double distilled water, and then dried in an oven. It was further treated with hydrogen peroxide (100 volumes) for about 24 h to remove all adhering organic particles and dried at 110 °C for 1 h in the vacuum oven. The material was grounded and sieved to desired particle sizes such as <106, 106–125, 125–180, 180–212, 212–250, 250–300, and >300 BSS mesh (British Standard Size).

2.2. Adsorption studies

The adsorption experiments were carried out in a batch process by using aqueous solution of Erythrosine with both the adsorbents and experiments were conducted to observe the effect of various parameters such as pH, temperature, particle size, amount of adsorbent, concentration, and contact time. Adsorption isotherms were recorded at equilibrium conditions for the concentration of dye over range 1×10^{-5} to 9×10^{-5} M at a fixed pH. The selected concentration was ascertained after a good deal of examination. Each adsorption study was made in a mechanically agitated 100 ml volumetric flask filled with 30 ml of a dye solution of desired concentration along with a known amount of adsorbent. When the equilibrium was established, supernatant was carefully filtered through Whatmann filter paper (no. 1) saturated with distilled water.

2.3. Kinetic studies

For kinetic studies, the batch technique was used due to its simplicity. A series of conical flasks of 100 mL capacity and containing a definite volume of solutions of Erythrosine of known concentrations were kept in a thermostat shaking water bath. After attaining the desired temperature, a known amount of the adsorbent was added to each flask and the flasks were allowed to agitate mechanically. At given time intervals the adsorbent was separated by filtration and the filtrate thus obtained was analyzed spectrophotometrically at 527 (λ_{max}) to determine the equilibrium concentration of the dye. The kinetic studies were also carried out under different adsorbate concentrations.

3. Results and discussion

3.1. Adsorbent characterization

For morphological characteristics scanning electron microscope (SEM) of adsorbent AC and ADM was carried out.

3.1.1. SEM analysis

The AC and ADM were analyzed by SEM is shown in Fig. 1. SEM is widely used to study the morphological features and surface characteristics of the adsorbent materials. In the present study, SEM photographs (Fig. 1) of AC and ADM reveals surface texture and porosity.

3.2. Effect of adsorbent dose

As the adsorbent dosage increases, the adsorbent sites available for the dye molecule also increases and consequently better adsorption takes place. In the present study, the adsorbent doses were varied from 0.06 to 0.72 g/l for activated carbon and 2.5–20 g/l for activated de-oiled mustard at fixed pH 3.5, temperature 30 °C and adsorbate concentration 5.0×10^{-5} M. It is apparent from Fig. 2(a) for activated carbon (b) for activated de-oiled mustard that the removal efficiency increased rapidly with increase in the concentration of the both adsorbent due to the greater availability of the

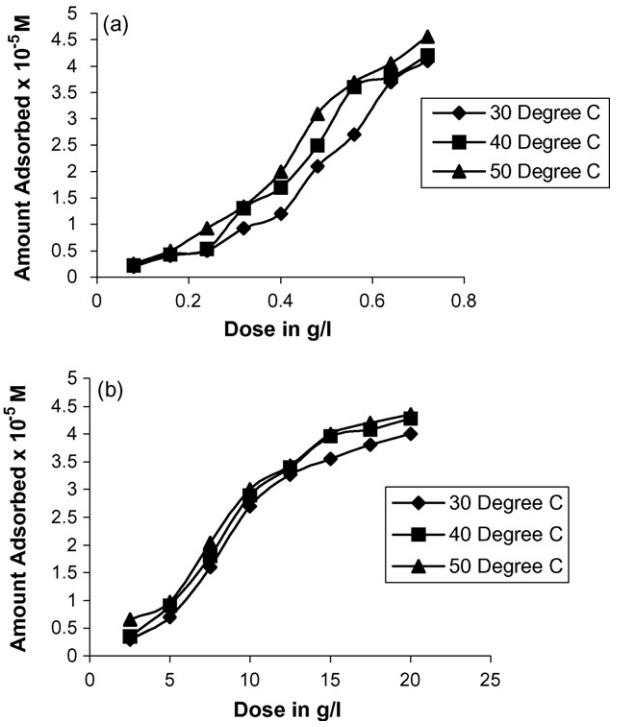

Fig. 2. Plots of adsorbent dose vs. amount adsorbed for Erythrosine at (a) AC and (b) ADM.

exchangeable sites or surface areas at higher concentrations of the adsorbent [40]. 0.56 g/l for AC and 12.5 g/l for ADM were found to be the optimum dose for treatment of Erythrosine dye.

3.3. Effect of substrate concentration

For adsorptive reaction, the rate varies directly with concentration of adsorbate. The extent of adsorption depends on the size, shape, mobility, and charge of molecules or ion present in solution. It has generally observed that adsorption decreases with increasing in initial dye concentration. For observing the effect of concentration range of adsorbate on removal of Erythrosine, the concentration range was varied from 1.0×10^{-5} to 9.0×10^{-5} M at a fixed dose of adsorbents 0.56 g/l for AC and 12.5 g/l for ADM and pH 3.5. It is apparent from Fig. 3 that adsorption increases from 1.0×10^{-5} to 5.0×10^{-5} M and then it becomes almost constant for

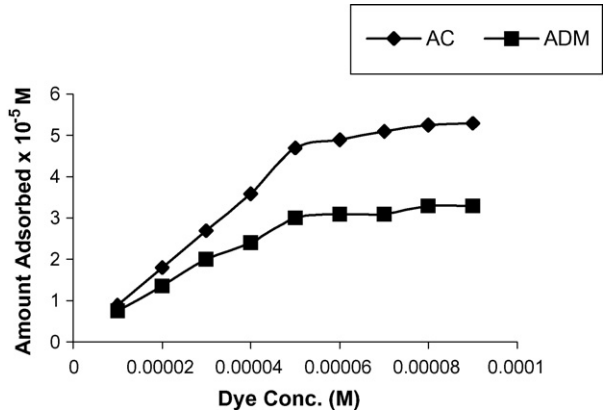

Fig. 3. Plots of concentration of adsorbate vs. amount adsorbed for Erythrosine for AC and ADM.

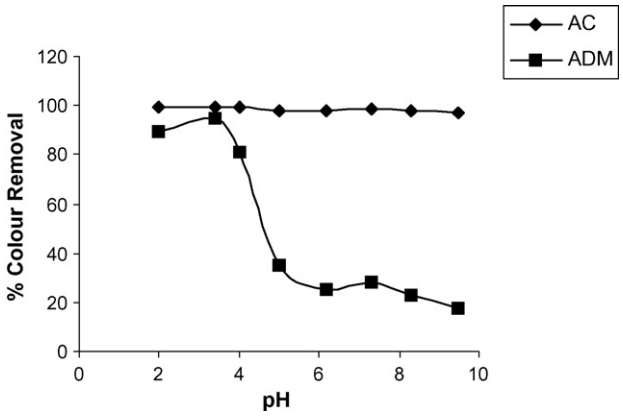

Fig. 4. Plots of pH vs. % colour removal for Erythrosine for AC and ADM.

both the adsorbents, indicating the optimum adsorption capacity of the adsorbent at 5.0×10^{-5} M.

3.4. Effect of pH

The experiments carried out at different pH shows that there is a change in the percent removal of dyes over the entire pH range from 2 to 9.4 as shown in Fig. 4. This indicates the strong force of interaction between the dye and both the adsorbents that, either H^+ or OH^- ions could influence the adsorption capacity. It is clear from Fig. 4 that the maximum uptake of dye takes place at pH 2.0–4.6 (99%) colour removal in case of activated carbon and for activated de-oiled mustard at pH 3.5 (95%) colour removal. It is also apparent that adsorption decreases with increasing pH for ADM. At pH 9.5 AC 97% while ADM adsorbs only 17.6%. AC is very efficient adsorbent in all media but in case of ADM it removes colour very efficiently in acidic medium and adsorption decreases with increasing pH.

3.5. Effect of particle size

The finer the particle size, the larger the surface area, which would in turn provide better contact between the adsorbate and adsorbent. Hence the adsorption capacity would be better. Smaller particles take a shorter time to equilibrate [41,42], Particles of <106 BSS mesh when compared to >300 BSS mesh showed better adsorption characteristics. The selected particle sizes were <106, 106–125, 125–180, 180–212, 212–250, 250–300, and >300 BSS mesh. Maximum adsorption for AC 94.8% and for ADM 84.3% occur at <106 BSS mesh and for both the adsorbents adsorption decreases with increasing mesh size of adsorbent at dye conc. 5.0×10^{-5} M, pH 3.5 and adsorbent dose 0.56 g/l for AC and 12.5 g/l for ADM.

3.6. Effect of contact time

The influence of contact time on colour removal by AC and ADM is presented in Fig. 5. It may be observed from Fig. 5 that the rate of removal of colour was rapid initially in case of activated de-oiled mustard. The rate leveled off gradually and then attained a more or less constant value (equilibrium) beyond which there was no significant increase in colour removal. The time required to attain equilibrium was 20 min for AC and 60 min for ADM.

3.7. Effect of temperature

The degree of adsorption depends on the temperature of the solid–liquid interface. The rate of adsorption was studied in the temperature range of 30–50 °C by withdrawing samples at specific

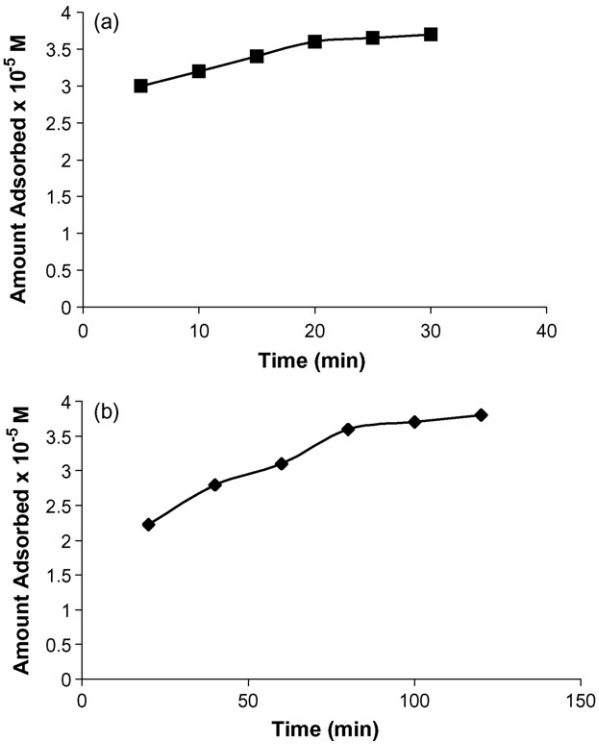

Fig. 5. Plots of contact time vs. amount adsorbed on the uptake of Erythrosine for (a) AC and (b) ADM.

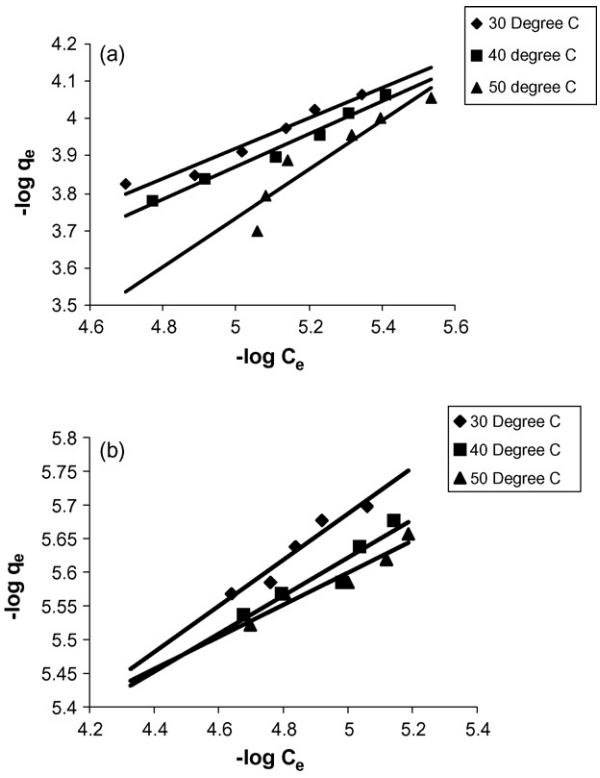

Fig. 7. Freundlich adsorption isotherm of Erythrosine for (a) AC and (b) ADM.

time intervals. Interestingly, it was observed that, at higher temperatures, the rate of adsorption was faster with both the adsorbents. The process was hence concluded to be endothermic and spontaneous. The adsorption follows the order 30 < 40 < 50 °C. The plots of the effect of temperature on the adsorption process are shown in Fig. 6.

4. Adsorption isotherms

The adsorption data were analyzed with the help of the following linear forms of Freundlich and Langmuir isotherms [43]. These isotherms are useful for estimating the total amount of adsorbent needed to adsorb a required amount of adsorbate from solution.

4.1. Freundlich isotherm

The performance of the adsorbent in dye removal from the aqueous solution has been studied using the Freundlich isotherm. The adsorption data for adsorption over AC and ADM were fitted into the linear form of Freundlich model. The logarithmic form of Freundlich model is given by the equation:

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

where q_e is the amount adsorbed (mol/g) and K_f and n are Freundlich constants related to the adsorption capacity and adsorption intensity respectively [44]. When $\log q_e$ was plotted against $\log C_e$, straight line with slope $1/n$ was obtained (Fig. 7(a) for AC and (b) for ADM respectively), which shows that the adsorption of dye on AC and ADM follows the Freundlich isotherm. Freundlich constants K_f and n are calculated for AC and ADM respectively, and the values at different temperatures are given in Table 1.

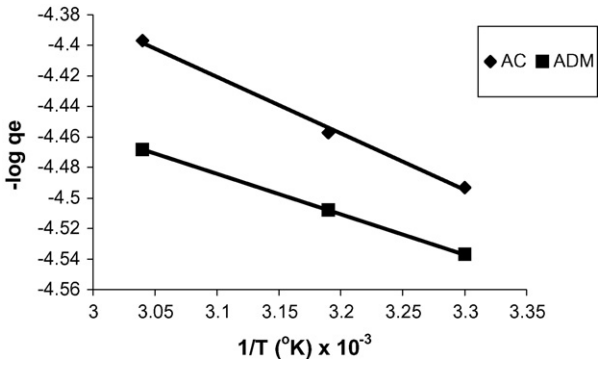

Fig. 6. Effect of temperature on adsorption of Erythrosine on AC and ADM.

4.2. Langmuir isotherm

Adsorption isotherm data have been described by the Langmuir adsorption isotherm [45]. The Langmuir isotherm has been used by many workers to study the sorption of a variety of compounds. The model assumes uniform energies of adsorption onto the surface and no transmigration of adsorbate in the plane of the surface. The results of the adsorption of dye on AC and ADM were fitted into Langmuir isotherm. The linear form of the isotherm was analyzed

Table 1
Freundlich constants for Erythrosine over AC and ADM

Temperature (°C)	Activated carbon			Activated de-oiled mustard		
	K_f	n	R^2	K_f	n	R^2
30	77.821	2.473	0.9611	9.408	2.918	0.9324
40	48.921	2.291	0.981	16.188	3.539	0.8945
50	2.6872	1.515	0.8704	25.532	4.192	0.9314

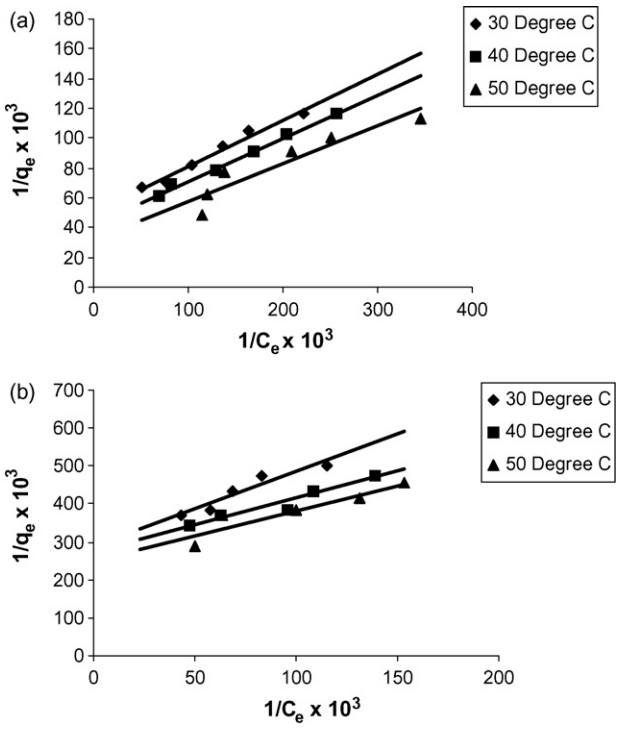

Fig. 8. Langmuir adsorption isotherms of Erythrosine for (a) AC and (b) ADM.

in the light of the model.

$$\frac{1}{q_e} = \frac{1}{Q_o} + \frac{1}{bQ_o}C_e \quad (2)$$

where q_e is the amount adsorbed (mol/g) and C_e is the equilibrium concentration of the adsorbate (mol/l). Q_o and b are the Langmuir constants related to maximum adsorption capacity and energy of adsorption, respectively. When $1/q_e$ is plotted against $1/C_e$, a straight line with slope $1/bQ_o$ is obtained (Fig. 8(a) for AC and (b) for ADM), which shows that the adsorption of Erythrosine follows Langmuir isotherm for both the adsorbents. The Langmuir constants Q_o and b are calculated and the values of these constants at different temperatures are tabulated in Table 2.

To confirm the favourability of the adsorption process the separation factor R_L was calculated by the following equation [46].

$$R_L = \frac{1}{1 + bC_o} \quad (3)$$

The values of R_L obtained were found between 0 and 1 and confirms the on-going adsorption process is favourable. The same method has already been adopted [47] to confirm the favourability of a Langmuir type of adsorption.

4.3. Thermodynamic parameters

The kinetics of the rate of adsorption was assessed at 30, 40, and 50 °C. The value of ΔH° determines whether a process is endothermic or exothermic. Here the value of ΔH° is found to be positive for both the adsorbents and hence the reaction involved is endothermic. Also the negative value of ΔG° indicates the spontaneous nature of adsorption of Erythrosine onto AC and ADM. Thermodynamic parameters ΔG°, ΔH° and ΔS° were calculated using the following equations, and the values are shown in Table 3. The positive values of ΔH° confirm the endothermic nature of adsorption [48].

$$\Delta G^\circ = -RT \ln b \quad (4)$$

$$\Delta H^\circ = -\frac{R(T_2 T_1)}{(T_2 - T_1)} \ln\left(\frac{b_2}{b_1}\right) \quad (5)$$

$$\Delta S^\circ = \frac{(\Delta H^\circ - \Delta G^\circ)}{T} \quad (6)$$

where b, b_1 and b_2 are Langmuir constants corresponding to the temperatures 30, 40 and 50 °C.

Table 3
Thermodynamic parameters of Erythrosine over AC and ADM

Adsorbent	$-\Delta G^\circ$ (kJ mol^{-1})	ΔH° (kJ mol^{-1})	ΔS° (J K^{-1} mol^{-1})
Activated carbon	12.81×10^3	9.747×10^3	74.44×10^{-3}
Activated de-oiled mustard	12.57×10^3	2.377×10^3	44.33×10^{-3}

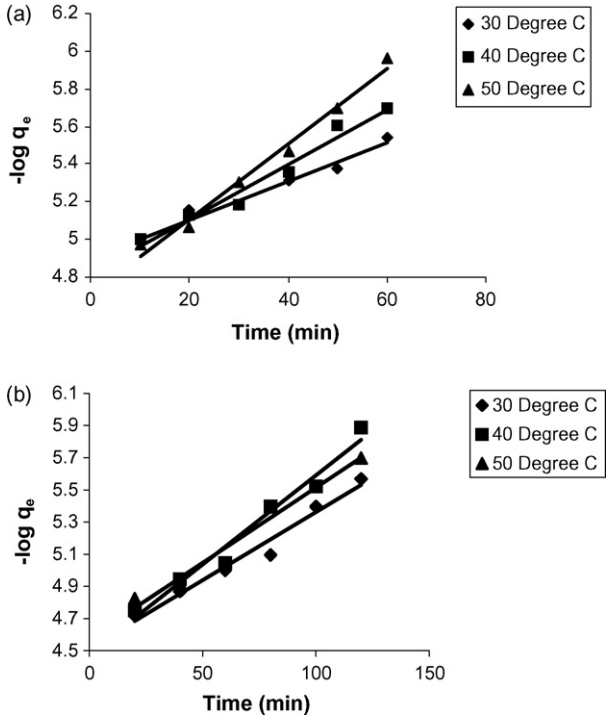

Fig. 9. Lagergren plots for Erythrosine adsorption at different temperatures for (a) AC and (b) ADM.

Table 2
Langmuir constants for Erythrosine over AC and ADM

Temperature (°C)	Activated carbon			Activated de-oiled mustard		
	b (mol g^{-1})	Q_o (l mol^{-1})	R^2	b (mol g^{-1})	Q_o (l mol^{-1})	R^2
30	161.57	0.0199	0.9766	147.3	3.463	0.9039
40	147.71	0.0234	0.9942	197.5	3.628	0.9284
50	131.539	0.0303	0.8782	191.9	3.969	0.8602

Table 4
Rate constants k_{ad} for Erythrosine over AC and ADM

Adsorbent	k_{ad}		
	30 °C	40 °C	50 °C
Activated carbon	23.26×10^{-3}	33.39×10^{-3}	46.06×10^{-3}
Activated de-oiled mustard	18.42×10^{-3}	25.56×10^{-3}	21.41×10^{-3}

4.4. Adsorption rate constant

The adsorption rate constant is determined from the first-order rate expression given by Lagergren rate equation [49].

$$\log(q_e - q_t) = \log\ q_e - k_{ad} \times \frac{t}{2.303} \quad (7)$$

where q_e and q_t are the amount of dye adsorbed (mol/g) at equilibrium and at time t (min), respectively, and k_{ad} the rate constant of adsorption (min^{-1}). For both the systems, the plots (Fig. 9(a) for AC and (b) for ADM) obtained for $\log(q_e - q_t)$ vs. t exhibit straight lines and confirm the adsorption process to follow first order rate kinetics in each case. The k_{ad} values evaluated, for each system, from the respective Lagergren plots are presented in Table 4.

5. Determination of activation energy for the adsorption process

The rate constant k_2 at different temperatures listed in Table 4 was then applied to estimate the activation energy of the adsorption of Erythrosine dye on to AC and ADM by the Arrhenius equation [50].

$$\ln\ k = \ln A - \frac{E_a}{RT} \quad (8)$$

where E_a, R and A refer to the Arrhenius activation energy, the gas constant and the Arrhenius factor, respectively. The slope of the plot of $\log k$ vs. $1/T$ (Fig. 10(a) for AC and (b) for ADM) was used to evaluate E_a and the value of E_a was found to be 7987.34 kJ mol^{-1} for AC and 3260.25 kJ mol^{-1} for ADM, which shows that both the adsorbents have strong binding sites.

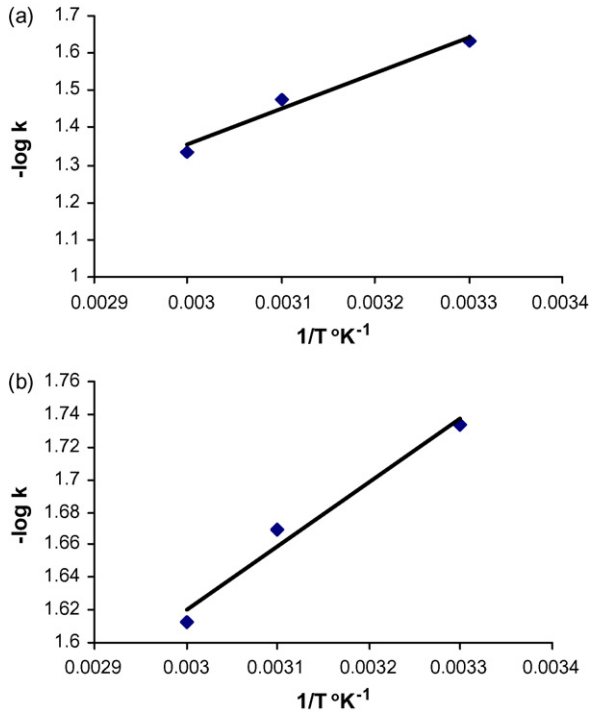

Fig. 10. Plot of $\log k$ vs. $1/T$ for (a) AC and (b) ADM.

6. Chemical oxygen demand

COD test is widely employed as means of measuring the pollution strength of domestic and industrial wastes. COD of initial coloured, treated filtrate of AC and ADM solutions was determined. Usual 2 h open reflux method [51] was applied for the COD determination and it was found that for the dye solutions COD values show a significant decrease (from 1600 to 176 mg/l and 218 mg/l when adsorbed over AC and ADM, respectively), indicating less toxicity of the treated products in comparison to original dye. The treated filtrate of AC and ADM with the percent COD removal efficiency of 88% and 86% indicates the usefulness of adsorptive treatment of wastewater.

7. Conclusion

- Adsorption of Erythrosine on the surface of adsorbent has also been found to be an efficient and economically cheap process. In the present study activated carbon and activated de-oiled mustard were used as an adsorbent for the removal of Erythrosine dye from an aqueous solution.
- The de-oiled mustard has very high adsorption capacity to remove the dye, with a monolayer adsorption capacity. The adsorption increases with the increase in amount of adsorbent. Freundlich and Langmuir equation agrees very well with the equilibrium isotherms.
- The first order kinetic model fits very well with the dynamical adsorption behaviour of Erythrosine dye. This oil mill waste residue could therefore be substituted in place of activated carbon as adsorbents due to its availability, high adsorption capacity and low cost.

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