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Adsorptive and desorptive studies on toxic dye Amaranth onto de-oiled mustard from wastewater

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ABSTRACT

De-oiled mustard, an oil mill waste, was treated as an adsorbent for Amaranth, an azo dye currently used in paper and textile industries. The presence of Amaranth in these effluents causes obvious environmental and health problems. Kinetic studies of adsorption of Amaranth to de-oiled mustard were conducted in batch conditions at 30°C. The paper incorporates effect of pH, temperature, amount of adsorbent, contact time, concentration of adsorbate, particle size on adsorption. The adsorption process followed a pseudo-first order model. The equilibrium process showed to be well described by both Freundlich and Langmuir models, at 30°C, 40°C and 50°C. Thermodynamic parameters like free energy (ΔG), enthalpy (ΔH) and entropy (ΔS) of the systems, respectively, were calculated by using Langmuir constant. The estimated values for ΔG^o were -8.027×10^3 and -1.5322×10^3 over activated carbon and activated de-oiled mustard at 303 K (30°C), indicate toward a spontaneous process. Desorption studies indicate that elution by dilute NaOH through the fixed bed of the adsorbents columns could be regenerated and a quantitative recovery of Amaranth can be achieved. A significant decrease in the COD values was observed, which clearly indicates that adsorption method offer a good potential to remove Amaranth from wastewater. Treated activated de-oiled mustard is an attractive candidate for removing organic dye Amaranth.

Keywords: Amaranth; Adsorption; Desorption; Kinetics; De-oiled mustard; COD

1. Introduction

Amaranth is a well-known azo dye, which is widely used for colouring textile materials, paper, wood, leather, etc. Though for a quite long time it was also used as colouring agent for foodstuff like jams, jellies, ketchup and cake decoration, but for the last few years the carcinogenicity and other toxic effects of the dye compelled authorities for its legal prohibition in many countries. It is now well established that a prolong intake of Amaranth can result into tumors, allergy, respiratory problems and birth defects [1–4] in the human being. Since Amaranth possesses exceptionally good solubility in water, hence its removal by common chemical treatments or by physical treatments like coagulation, froth floatation, etc. is not easy. Generally biological aerobic wastewater systems are not successful for decolourization of majority of dyes [5]. Among the physicochemical processes, adsorption technology is considered to be one of the most effective and proven technology having potential application in water and wastewater treatment, a tested method for the removal of hazardous organic compounds [6]. Adsorption is a rapid phenomenon of passive sequestration and separation of adsorbate from aqueous/gaseous phase on to solid phase. Textile dye colour removal by adsorption onto activated carbon has proven to be highly efficient and reliable.

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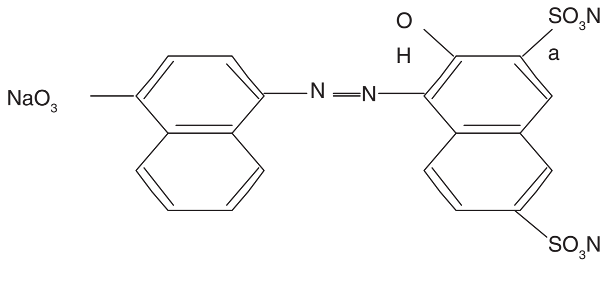
Possessing high specific surface area, activated carbon frequently exhibits high removal efficiency for most dissolved compounds. Inspite of this it suffers from few disadvantages. Activated carbon is quite expensive, and its regeneration produces additional effluent and results in considerable loss (10–15%) of the adsorbent. As far as removal of hazardous substances from wastewater is concern, during the last few years the focus of the research is to exploit the use of industrial and agricultural waste products as potential adsorbents. Literature survey reveals that a large number of such waste products like bagasse fly ash [7], bottom ash [8], de-oiled soya [9], rice husk and gram husk [10,11], etc. have been utilized as potential adsorbent. Gupta et al. and Mittal et al. [12–24] have also utilized various adsorbents for the removal of dyes from wastewater. Our laboratories are also contributing in this direction with adsorption, photocatalytic and electrochemical methods for the removal of some toxic textile and food dyes [25–28].

The object of the present investigations has been to evaluate the efficiency of removal of Amaranth using activated carbon (AC) and activated de-oiled mustard (ADM). The de-oiled mustard is the processes out waste material from oil mill, which is obtained after extracting all possible nutrients of de-oiled mustard [29,30]. India is one of the leading producers of the mustard crop. Thus, use of de-oiled mustard, as an adsorbent is a thoughtful attempt for its utilization for the mankind. So from economical point of view the use of this adsorbent is very beneficial and effective in removing Amaranth from the aqueous solutions and their performance was evaluated against activated carbon.

2. Materials and methods

The dye under consideration is Amaranth (A) ($C_{20}H_{11}N_2Na_3O_{10}S_3$; Mol. wt. 604.6), which is highly water soluble dye of azo group was obtained from M/s Merck. The stock solution and all other solutions of Amaranth were prepared in double-distilled water, and the same was used for the necessary dilution. Aqueous solutions of Amaranth with different concentrations were prepared from 0.01 M stock solution. All reagents used in the present work were of analytical grade. The adsorbent de-oiled mustard was collected from a local oil mill, while AC was purchased from M/s Merck and used as received. Measurements of pH of the solutions were carried out on a digital pH meter DB 1011, fitted with a glass electrode. A 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

The adsorbent material, activated carbon was used as received. The other material, deoiled mustard, was cleaned, thoroughly washed with distilled water, and then dried in an oven. Thereafter, it was treated with hydrogen peroxide (30%) at 60°C for 24 h to oxidize the adhering organic matter. The resulting material was washed with double distilled water and filtered and again dried to 100°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, >300 BSS mesh (British Standard Size). Finally, granules of ADM thus obtained were stored in separate vacuum desiccators until required.

2.2. Adsorption studies

Adsorption studies were performed by the batch technique at 30°C, 40°C and 50°C, temperatures. Adsorption isotherms recorded at a fixed pH range (2.0–9.5) over the concentration range 1.0×10^{-5}–9.0×10^{-5} mol/l of Amaranth solutions, prepared by diluting 0.01 mol/l of stock solution in a series of 100 ml graduated conical flask containing 30 ml of solution of each concentration. Adsorption was achieved by adding a known amount of each adsorbent of a specific sieve size into the dye solution of known concentration and pH, and the conical flask were agitated intermittently. Once the equilibrium was established, supernatant liquid was filtered off using Whatmann filter paper No. 42 and uptake of the dye was determined spectrophotometrically by measuring the absorbance at 520 nm.

2.3. Kinetic studies

Kinetic investigations were carried out by batch technique, because of its simplicity. Thirty-milliliter solution of Amaranth of known concentration and 0.5 g/l for (AC) and 6.66 g/l for (ADM) were taken in airtight 100 ml conical flasks. Keeping the flasks in a water bath, maintained at desired temperature (30°C), mixture was mechanically agitated. After a definite interval of time, the solution of the flasks was filtered and filtrate of each was analyzed for the uptake of dye by using a UV–Vis spectrophotometer set at wavelength of 520 nm, maximum absorbance. The dye uptake q (mol/g) was determined as follows:

$$Q = \left(C_0 - C\right) V/W \tag{1}$$

where C_0 and C are the initial and final dye concentrations (mol/l), respectively, V is the volume of solution (l), and W is the sorbent weight (g).

Calibration experiments were carried out to exclude the experimental mistake raised from sorption of Amaranth on wall of the glass vessels. All the experiments were carried out in triplicate and the mean values are presented.

2.4. Batch equilibrium studies

The procedures of equilibrium tests were basically identical to those of kinetic experiments. The aqueous samples were taken at preset time intervals, and the concentrations of dye were similarly measured.

2.5. Desorption studies

Regeneration is the most significant aspect of the adsorption study. Continuing emphasis was being placed on waste minimization, recovery and reuse. To assess the practical utility of the adsorbents, bulk adsorption were done by employing column operations. Adsorption column chromatography method [31] is adopted. Suitable adsorbent containing adsorbed dye is packed in a column, and appropriate eluent is passed through the column repeatedly at intervals till maximum colour is eluted out. The column operations was carried by using corning glass column of 30 cm length and 1 cm internal diameter, after packing it with a known amount, 300 mg AC of 125–180 BSS mesh and 400 mg of ADM of 106–125 BSS mesh, on a glass wool support. The weighed adsorbent was made into slurry with water and kept overnight and then fed slowly into column, displacing the heel of water, to avoid air entrapment. The slurry of the adsorbent was prepared in water only and kept overnight before feeding. The column was then loaded with a dye solution of appropriate concentration, which was allowed to percolate downwards under gravitational force at a flow rate of 0.5 ml/min. Then the amount of dye desorbed was estimated spectrophotometrically. After about 90% of exhaustion column operations were shut down. Thus desorption studies help in the recycling and regeneration of the spent adsorbent and the dye.

3. Results and discussion

3.1. Adsorbent characterization

For morphological characteristics SEM of adsorbent AC and ADM was carried out. The AC and ADM were analysed by scanning electron microscope (SEM) as 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 of AC and ADM reveals surface texture and porosity. The surface area of AC and ADM as calculated by Brunauer–Emmett–Teller (BET) method is 929.7 m^2/g and 326.5 m^2/g, respectively. Scanning electron microscopy was performed using a Philips SCI quanta 400 instrument.

3.2. Effect of adsorbent dose

The rate of uptake of adsorbate was studied as a function of the mass of adsorbent. As the adsorbent dose increases, the adsorbent sites available for the dye molecules also increase and consequently better adsorption takes place [32]. To optimize the adsorbent dose for the removal of Amaranth from its aqueous solutions, adsorption was carried out with different adsorbent dosages at different temperatures. The dose of adsorbent was varied from 0.1 to 0.7 g/l for AC and from 1.66 to 10 g/l for DM at fixed pH 3.4, and adsorbate concentration 6.0 × 10^{-5} M. As shown in Fig. 2(a) for AC and (b) for ADM, the adsorption increases with increasing amount of adsorbent from 0.1 to 0.5 g/l in case of AC whereas from 1.66 to 6.66 g/l for ADM at initial temperature and thereafter it remains constant. This is due to the unavailability of the adsorbate, the percentage adsorption remains constant. Hence all further studies were carried out using optimum dose 0.5 g/l for AC and 6.66 g/l for ADM.

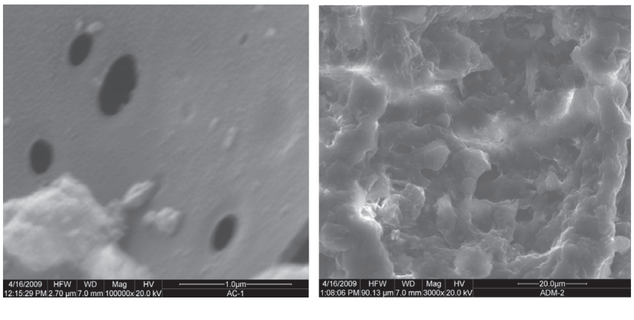
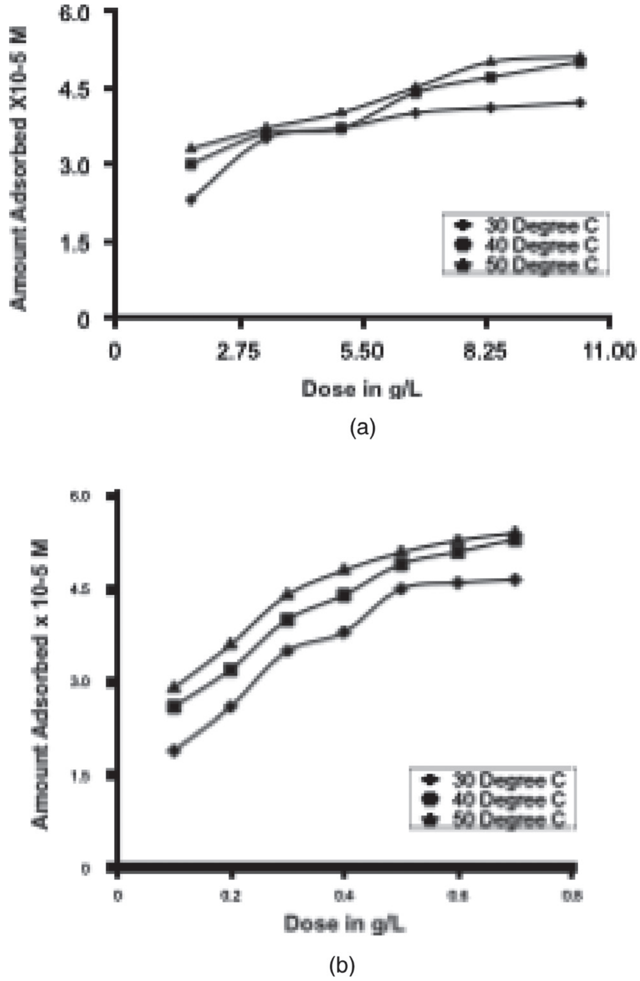


Fig. 1. SEM micrographs of (a) AC and (b) ADM adsorbents.



(a)

(b)

Fig. 2. Effect of amount of adsorbent for the removal of Amaranth (6×10^{-5} M) by (a) AC – 0.5 g/l and (b) ADM – 6.66 g/l at pH 3.4 and different temperatures.

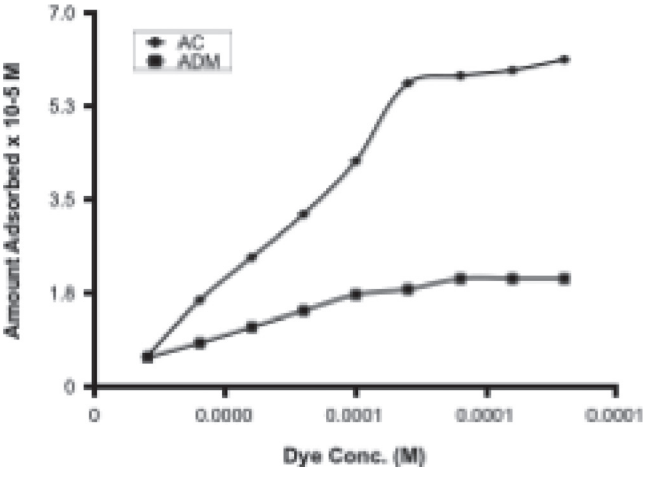


Fig. 3. Effect of initial dye concentration for the removal of amaranth by (a) AC – 0.5 g/l and (b) ADM – 6.66 g/l at 30°C and pH 3.4.

3.3. Effect of adsorbate concentration

There is a direct relationship between initial dye concentration and removal rate. The adsorption experiments were carried out in concentration range 1×10^{-5} to 9×10^{-5} M. The results are given in Fig. 3. Figure indicates that the adsorption of the total amount of dye increases with an increase in the dye concentration from 1×10^{-5} M to 6×10^{-5} M in the solution, for AC, and thereafter it is more or less practically constant, whereas in case of ADM it remains constant after 6×10^{-5} M. It shows that removal of dye is dependent upon the concentration of dye in a solution. In general, the percentage removal decreases from 78.79% to 61.79% for AC and 62.65% to 21.79% for ADM with the increase in dye concentration. The initial rate of adsorption was greater for a higher initial dye concentration because as resistance to dye uptake decreased, the mass transfer driving force increased, in agreement with the work of other researcher (Wang et al., 2005).

3.4. Effect of pH

The pH is one of the important factors controlling the adsorption of dye on to adsorbent. To study the influence of pH (Fig. 4) on the adsorption capacity of AC and ADM for Amaranth, experiments were carried out using different initial solution pH values, varying from (2.0–9.5). For AC, it was observed that the percentage of dye removal was not affected by pH variation. The uptake of the dyes was nearly 100% for all pH values. For ADM it may be observed from the figure that the dye uptake was higher at lower pH and as the pH of the dye solution increased, dye uptake decreased considerably from 80.3% to 17.6%. At acidic pH, the positively charged species start dominating and surface tends to acquire a positive charge, while the adsorbate species are still negatively charged. As the adsorbent surface is positively charged the increasing electrostatic

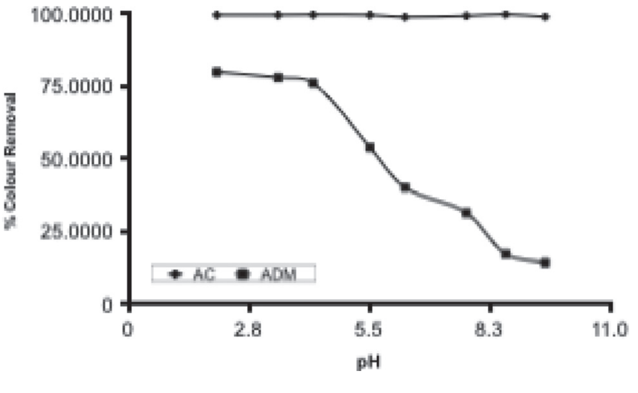


Fig. 4. Effect of pH for the removal of amaranth (6×10^{-5} M) by (a) AC – 0.5 g/l and (b) ADM – 6.66 g/l at 30°C.

attraction between negatively charged adsorbate species and positively charged adsorbent particles would lead to increased adsorption of Amaranth dye [33]. The low sorption uptake at a higher pH range may be due to competitive adsorption of OH^- ions and dye anions [34]. Hence, all the succeeding investigations were performed at pH 3.4 for both the adsorbents.

3.5. Effect of particle size

The adsorption rate is expected to vary as the reciprocal of the dm of the adsorbent particle for a given total weight of adsorbent. In present investigations the adsorbent of different particle sizes <106, 106–125, 125–180, 180–212, 212–250, 250–300, >300 BSS mesh were taken at fixed dose 0.5 g/l for AC and 6.66 g/l for ADM and pH 3.4. It is clear from Fig. 5, the uptake of dye increases with decreasing adsorbents particle size for both the adsorbents. This relationship clearly demonstrates the advantage of using powdered adsorbent rather than the granular form from a kinetic viewpoint, as it indicates that external transport limits the rate of adsorption in these cases [35]. Maximum adsorption for AC 90.23% and for ADM 71.10% was observed at <106 BSS mesh.

3.6. Effect of temperature

The temperature effect on decolourization rate is significant. It is apparent from Fig. 6 that adsorption for AC and ADM increases with increase in temperature in the order of 30°C < 40°C < 50°C. This may be due to the fact that with increase in temperature, rate of diffusion of the adsorbate molecules across the external boundary layer and in the internal pores of the adsorbent particle increases, owing to the decrease in the viscosity of the solution. In addition, changing the temperature will change the equilibrium capacity of the adsorbent for a particular adsorbate [36].

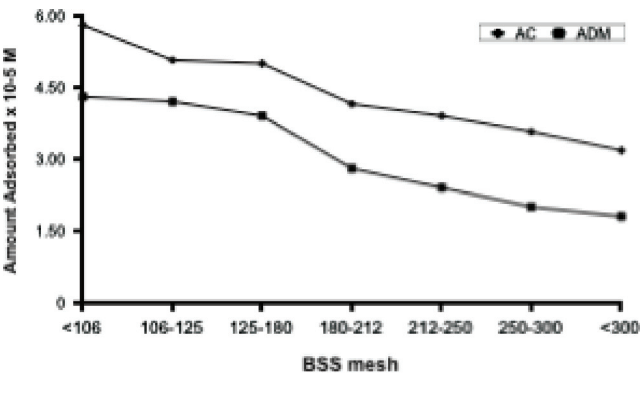


Fig. 5. Effect of particle size for the removal of Amaranth by (a) AC – 0.5 g/l and (b) ADM – 6.66 g/l at 30°C and pH 3.4.

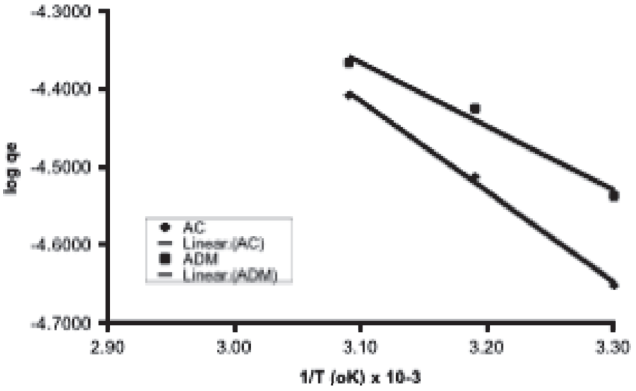


Fig. 6. Effect of temperature for the removal of Amaranth by (a) AC – 0.5 g/l and (b) ADM – 6.66 g/l at pH 3.4.

4. Adsorption isotherms

Adsorption data for a wide range of adsorbate concentration and adsorbent doses have been analyzed using Langmuir and Freundlich isotherms in order to find the adsorption capacity of AC and ADM. The linear plots of Langmuir isotherms determine whether the ADM can be used as low cost adsorbent by calculating the separation factor R_L by using Langmuir equation. The linear graph of Langmuir isotherms also represents the correlation coefficient and the value of intercept that is presented in Table 1. The linear plots of log q_e and log C_e show the strength of adsorption capacity (Fig. 7(a) for AC and (b) for ADM). The applicability of the Langmuir isotherm with the R_L values [37] in the range of 0–1 indicates that the adsorption process is favourable (Fig. 8(a) for AC and (b) for ADM). The energy of adsorption is constant and there is no transmigration of adsorbate in the plane of the surface of adsorbents. The Freundlich isotherm describes equilibrium on a heterogeneous surface where energy of the adsorption was not equivalent for all adsorption sites, thus allowing multi-layer adsorption. A more heterogeneous the surface will result in an $1/n$ value closer to zero [38]. Values of k_f and n were calculated from the slope and intercept of the Freundlich plots respectively (Table 2). The magnitude of the exponent 'n' gives an indication of the favourability and k_f is the capacity of the adsorbent/adsorbate system. Result from this experiment shows the n values ranging between 1 and 10, indicating beneficial adsorption.

5. Thermodynamic parameters

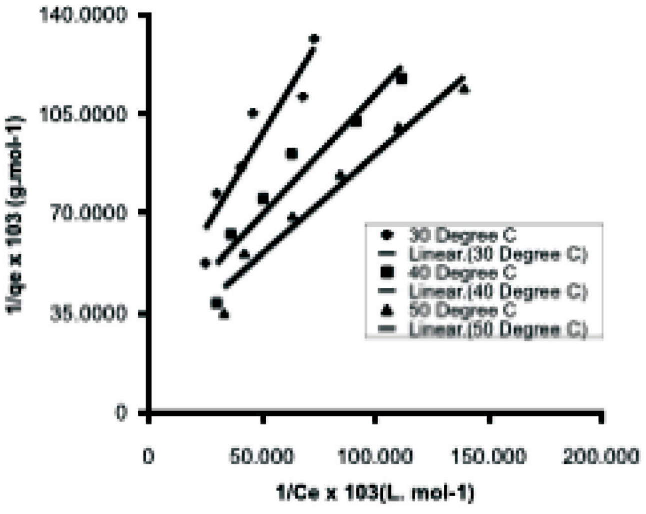
Different thermodynamic parameters, Gibb's Free energy (ΔG^o), enthalpy (ΔH^o) and entropy (ΔS^o) of the process were calculated by employing following equations:

$$\Delta G^\circ = -RT \ln b \qquad (2)$$

$$\Delta H^\circ = -R(T_2 T_1) / (T_2 - T_1) \ln(b_2/ b_1) \qquad (3)$$

Table 1
Freundlich constants for the adsorption of Amaranth over AC and ADM at ph 3.4 and different temperatures

Temperature	Activated carbon			Activated de-oiled mustard		
	b (mol g^{-1})	$Q°$ (l mol^{-1})	R^2	b (mol g^{-1})	$Q°$ (l mol^{-1})	R^2
30°C	24.192	0.0311	0.8808	1.8370	0.1657	0.9511
40°C	32.33	0.0364	0.9081	9.035	0.0562	0.851
50°C	30.41	0.0471	0.9607	5.437	0.1119	0.9074



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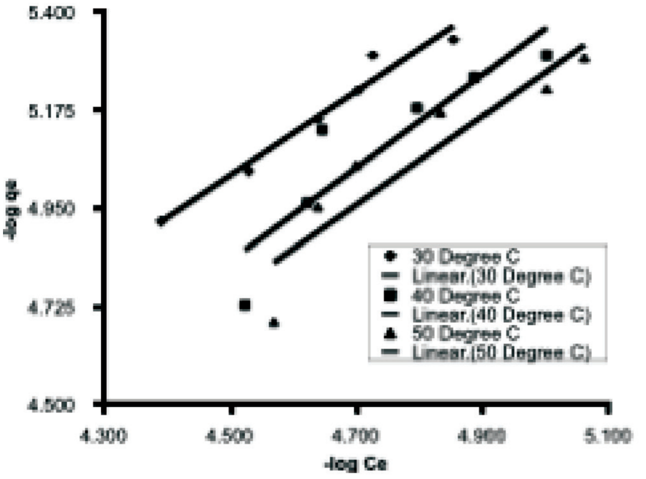
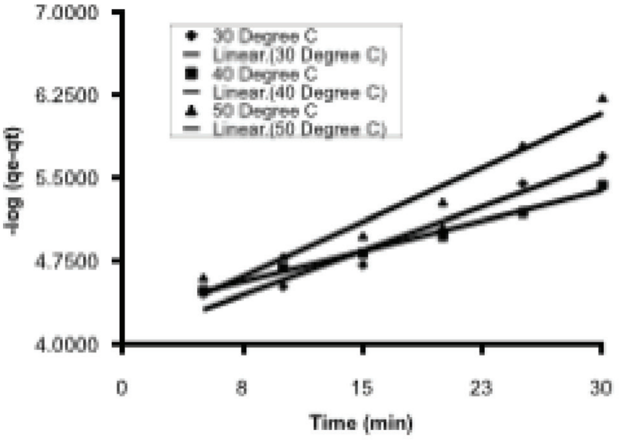


(b)

Fig. 7. Freundlich adsorption isotherms for adsorption of Amaranth over (a) AC (b) ADM at pH 3.4.



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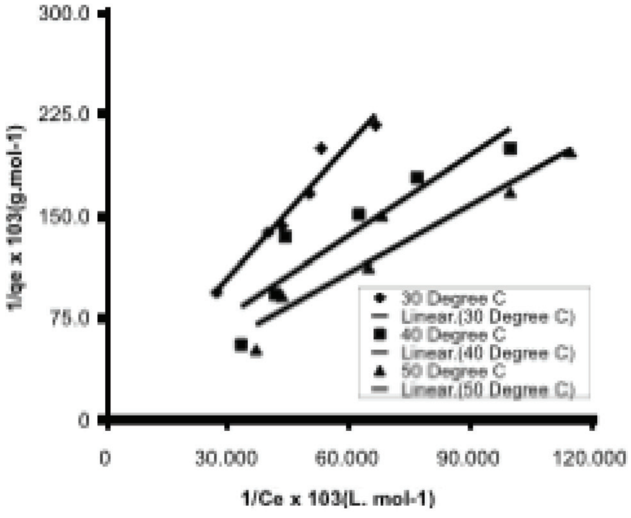


(b)

Fig. 8. Langmuir adsorption isotherms for adsorption of Amaranth over (a) AC (b) ADM at pH 3.4.

$$\Delta S^\circ = (\Delta H^\circ - \Delta G^\circ) / T \quad (4)$$

where b, b_1, b_2 are the equilibrium constants at different temperatures, which are gathered from the slopes of straight lines obtained in case of Langmuir adsorption isotherms at different temperatures. The values of the thermodynamic parameters obtained by above mentioned equations are presented in Table 3. Negative value of ΔG^o indicates feasible and spontaneous nature of the

Table 2
Langmuir constants for the adsorption of Amaranth over AC and ADM at pH 3.4 and different temperatures

Temperature	Activated carbon			Activated de-oiled mustard		
	k_f	n	R^2	k_f	n	R^2
30°C	21.677	1.7761	0.8567	4.696	0.9677	0.9643
40°C	4.8696	1.4907	0.9072	1.0548	1.0683	0.815
50°C	1.9177	1.3586	0.9605	1.5463	1.5463	0.8426

Table 3
Thermodynamic parameters for the uptake of Amaranth over AC and ADM at pH 3.4.

Adsorbent	ΔG° (kJ mol^{-1})	ΔH° (kJ mol^{-1})	ΔS° (kJ mol^{-1})
Activated carbon	8.027×10^3	5.12×10^2	43.38×10^{-3}
Activated de-oiled mustard	1.5322×10^3	42.682×10^3	145.92×10^{-3}

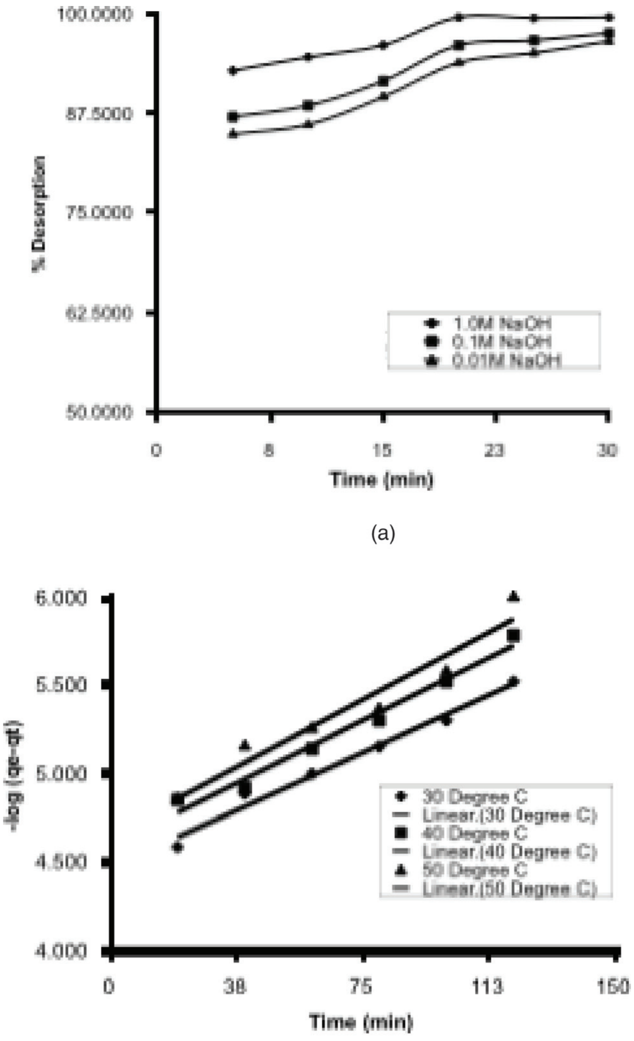
ongoing adsorption. It is also observed that in each case, ΔG^o value decreases with the increasing temperature, indicating thereby greater adsorption at higher temperature. Endothermic nature of the process was once again confirmed by obtaining positive values of ΔH^o.

6. Dynamic modeling

In order to examine the controlling mechanism of the adsorption process, pseudofirst order and pseudo second order equations were used to test the experimental data. A simple kinetic analysis of adsorption is the pseudo first order rate expression of the Lagergren equation [39] was employed:

$$\log(q_e - q_t) = \log q_e - k_{ad} \times t \,/\, 2.303 \tag{5}$$

where q_e and q_t are the amount adsorbed at equilibrium and time t, respectively. The time versus log $(q_e - q_t)$ plot (Fig. 9(a) for AC and (b) for ADM) gave straight lines at all the temperatures, which confirm the first order nature of the process. The slope of each straight line gave value of the rate constant, k_{ad} at that temperature. An increase in values of k_{ad} further confirms the increase in uptake of dye by increasing temperature. The k_{ad} values evaluated, for each system, from the respective Lagergren plot are presented in Table 4. The correlation coefficients for the pseudo second order kinetic model are all <0.95, indicating a poor pseudo second order fit to the experimental data.



(a)

(b)

Fig. 9. Lagergren plots for adsorption of Amaranth over AC and ADM at pH 3.4 and different temperatures.

7. Recovery of toxic dye Amaranth

Recovery of the adsorbed Amaranth and regeneration of column was made by eluting dilute NaOH solution (Fig. 10). Three different concentration of NaOH, i.e., 1.0 M, 0.1 M and 0.01 M which was passed at a flow

Table 4
Values of rate constant for the uptake of Amaranth over AC and ADM at pH 3.4

Adsorbent	k_{ad}		
	30°C	40°C	50°C
Activated carbon	122.28×10^{-3}	84.25×10^{-3}	150.6×10^{-3}
Activated de-oiled mustard	20.33×10^{-3}	21.87×10^{-3}	23.49×10^{-3}

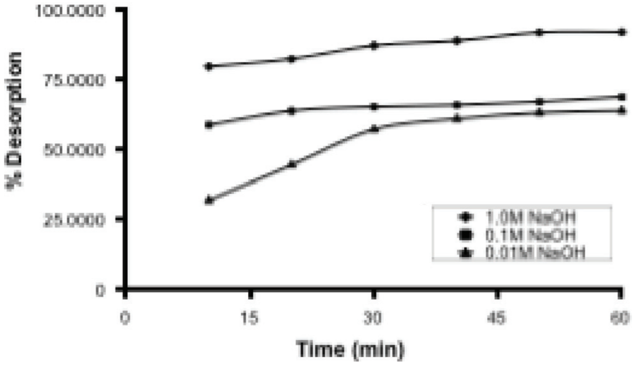


Fig. 10. Desorption profiles of Amaranth of AC and ADM using different concentrations of NaOH.

rate of 5 ml/min and fractions collected after every 10 min, which were analyzed spectrophotometrically. It has been observed from Fig. 10(a) for AC and (b) for ADM that maximum dye 99% desorbed from AC and about 92.2% dye desorbed from ADM with 1.0 M NaOH. After complete recovery of the dye the column was finally washed with the hot water.

8. Chemical oxygen demand (COD)

The chemical oxygen demand test is widely used to measure the organic strength of wastewater. The test allows the measurement of waste in terms of the total quantity of oxygen required for the oxidation of organic matter to CO_2 and water. The COD of the dye solution before and after the treatment was estimated. Usual 2 h open reflux method [40], was applied for the COD determination and the treated solution showed a significant decrease in COD value of the initial colour solution from 2180 mg/l to 127 mg/l and 212 mg/l, respectively, indicating the lower toxicity of the solutions left after adsorption treatment. Fig. 11 represents the effect of initial COD conc. on % COD reduction by commercial AC and ADM at the optimum pH, adsorbent dose and the contact time. The comparison in trend of % COD reduction by ADM with respect to AC under this condition is depicted in Fig. 11. The reduction in COD values of the treated dye solution indicates less toxicity of the treated products in comparison to original dye.

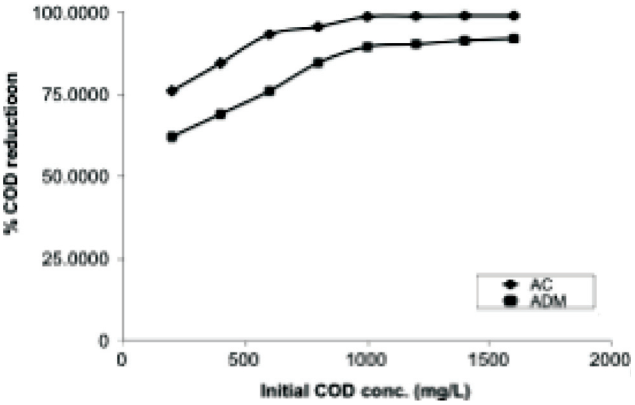


Fig. 11. Effect of initial COD concentration on % COD reduction of Amaranth by AC and ADM.

9. Conclusion

The present work is an attempt to develop a versatile, economic and reliable method for the removal of toxic dye, Amaranth from wastewater. Preliminary batch studies indicate that de-oiled mustard can adsorb almost 21.79% to 62.65% of the Amaranth from its aqueous solutions in the concentration range 1.0×10^{-5} M to 9.0×10^{-5} M, at 30°C. The adsorption data also confirm the validity of Freundlich and Langmuir adsorption isotherm models. At all temperatures, the adsorption process is feasible, spontaneous and endothermic in nature. The pseudo first order kinetic model fits very well with the high correlation coefficients (>0.95). Desorption studies indicate that by eluting dilute NaOH through the fixed bed of the adsorbents their columns can be regenerated and a quantitative recovery of Amaranth can be achieved. The percentage removal of dye was almost similar in both cases. Being a waste product, the use of activated de-oiled mustard, as adsorbent would also solve their disposal problem. In view of all these findings, it may be concluded that the developed adsorbent is very useful, economic, and reproducible for the removal of Amaranth. Thus, it may be safely concluded that ADM is quite economic than available commercially adsorbents for controlling the water pollution by dyes. Many types of sorbent are available for sorption. However, activated carbon is found to be the most popular adsorbent. Commercial activated carbons were prepared from various sources such as charred bone, wood and partial gasification. Yet, it is quite expensive and ineffective against many dyes. Therefore, studies have been carried out to investigate potential sources of raw material that could produce activated products. Wastes from agrobased industries are of attention mainly because of their abundance. Production of adsorbents from this source may reduce the cost of wastewater treatment, and at the same time open new market for low-cost agricultural by-products. In this study, de-oiled mustard obtained from oil mill has been chosen as the raw material to be activated. The composition of de-oiled mustard consists in 43% proteins, 2.05% oil, 1.22% allylisothiocyanate (AIT) and 2.75% phytic acid [41]. The availability of this source together with its characteristics has made de-oiled mustard a suitable alternative for activated carbon production. The result of this study may provide useful information on the efficiency of ADM for colour removal.

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