

# Equilibrium and Thermodynamic Studies on the Adsorption of the Dye Rhodamine-B onto Mustard Cake and Activated Carbon

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Mustard cake, obtained from local oil mills, has been characterized and used as an inexpensive and effective adsorbent for the removal of rhodamine-B dye from wastewater. The influence of various factors on the adsorption capacity has been studied by batch experiments. The optimum contact time to reach equilibrium was found to be 6 h. Maximum decolorization took place at pH 2.30. The optimum adsorbent dose was 5 g·L<sup>-1</sup> of particle size < 106 μm. The ongoing adsorption validates both the Langmuir and the Freundlich adsorption isotherms at temperatures of (40, 50, and 60) °C. Thermodynamic parameters indicate the feasibility of the process. The desorption profile revealed that a significant portion (80 %) of rhodamine-B could be desorbed by using 50 % ethanol as eluting agent. Desorption studies indicated the possibility of recycling and regeneration of both the adsorbent and the dye.

## 1. Introduction

Effluents from the textile, paper, and pulp industries contaminate the environment. Before discharging dyeing industry wastewater into natural water streams, it needs to be treated. The adsorption technique is the most widely used process for this purpose.<sup>1–5</sup> Basic dyes are the brightest class of soluble dyes used by textile industries. Rhodamine-B is a red dye used to dye wool, silk, and tanin mordanted cotton. Among the various adsorbents, activated carbon is widely used.<sup>6</sup> However, owing to the high cost of activated carbon and difficulties associated with its regeneration, several researchers have searched for alternate inexpensive materials for the removal of different pollutants.<sup>7–21</sup> Alternative adsorbents investigated are wood,<sup>13</sup> lignite and sphagnum peat,<sup>14,22–24</sup> fly ash,<sup>6,25–27</sup> rice husk,<sup>28–33</sup> and so forth.

The present study deals with the removal of rhodamine-B (see the structure) from water effluents on a low-cost adsorbent, mustard cake, obtained from local oil mills, and its efficiency was compared with activated carbon. Desorption profiles of the adsorbed dye from mustard cake were also studied.

## 2. Materials and Methods

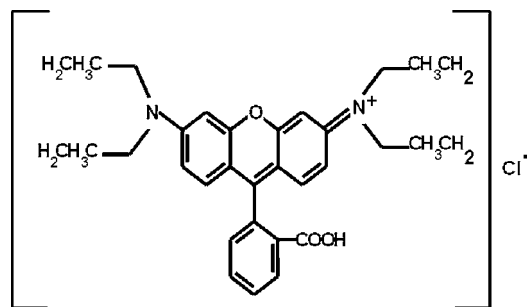
**2.1. Chemicals.** In the present investigation water-soluble rhodamine-B (molecular formula C<sub>28</sub>H<sub>31</sub>ClN<sub>2</sub>O<sub>3</sub>, molecular weight 479.02 g·mol<sup>-1</sup>) was obtained from Merck, and a 0.01 M stock solution was prepared in double-distilled water. Solutions of desired concentrations of the adsorbate were prepared from the stock solution, where double-distilled water was used for necessary dilutions. All reagents used were of analytical grade.

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Adsorbent activated carbon was also purchased from Merck, and mustard cake was collected from local oil mills.

**2.2. Preparation of Adsorbent.** Mustard cake was crushed thoroughly, washed with distilled water, and then dried in an oven. The dried material was then treated with hydrogen peroxide solution and left for 24 h to oxidize adhering organic impurities. Then the residual solution was dried at 110 °C for 1 h in a vacuum oven. The dried adsorbent was ground and sieved to desired particle sizes of (< 106, 106 to 125, 125 to 180, 180 to 212, 212 to 250, 250 to 300, and > 300) μm mesh size and stored in separate vacuum desiccators for further studies.

Scanning electron microscopy (SEM) was performed using a Philips SCI quanta 400 instrument. The zero point charge of the activated deoiled mustard was determined by potentiometric mass titrations (PMT).<sup>34</sup>

The physical characteristics of the mustard cake prepared as adsorbent are 43 % proteins, 2.05 % oil, 1.22 % allylthiocyanate (AIT), and 2.75 % phytic acid.<sup>35</sup>

**2.3. Experimental Procedure.** All pH measurements were carried out with a Decibel DB 1011 digital pH meter, fitted with a glass electrode. Absorbance measurements were recorded spectrophotometrically on a Spectronic 20 D+ Thermospectronic spectrophotometer over a wavelength range of (200 to 800) nm.

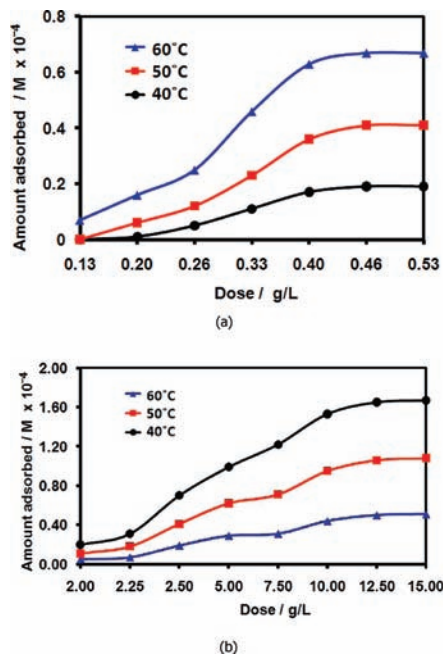


Figure 1. Plot of adsorbent dose vs adsorption of (a) rhodamine-B–charcoal and (b) rhodamine-B–mustard cake at different temperatures.

**2.4. Adsorption Studies.** Adsorption studies of charcoal and mustard cake were performed by a batch technique at (40, 50, and 60) °C. A series of Erlenmeyer flasks containing equal volumes (30 mL) of adsorbate solution at varying concentrations were employed at the desired pH. An optimized amount of adsorbent of a particle size of (106 to 125)  $\mu\text{m}$  was then added in each flask and intermittently agitated for a suitable contact time of 60 min and 6 h for charcoal and mustard cake, respectively. Once the equilibrium was established, supernatant liquid was filtered off using Whatmann filter paper no. 1, and uptake of the dye was determined spectrophotometrically at  $\lambda_{\text{max}}$  of 552 nm.

**2.5. Desorption Studies.** Desorption studies were carried out on a column. Regeneration is the most significant aspect of the adsorption study. A suitable adsorbent (mustard cake) containing adsorbed dye (rhodamine-B) was packed in a column, and then a suitable eluent was passed through the column repeatedly at intervals until the maximum color was eluted out. Then the amount of dye desorbed was estimated spectrophotometrically. Thus desorption studies help in the recycling and regeneration of the spent adsorbent and the dye.

### 3. Results and Discussion

**3.1. Characterization of Adsorbent.** For morphological characteristics, an SEM analysis of adsorbent charcoal and mustard cake was carried out. The charcoal and mustard cake were analyzed by SEM.<sup>14</sup> SEM is widely used to study the morphological features and surface characteristics of adsorbent materials. The surface area of charcoal and mustard cake as calculated by the Brunauer–Emmett–Teller (BET) method is  $929.7 \pm 2.1 \text{ m}^2 \cdot \text{g}^{-1}$  and  $326.5 \pm 3.4 \text{ m}^2 \cdot \text{g}^{-1}$ , respectively. Scanning electron microscopy was performed using a Philips SCI quanta 400 instrument.

**3.2. Effect of Adsorbent Dose.** To optimize the adsorbent dose for the removal of rhodamine-B from its aqueous solutions, adsorption was carried out at temperatures of (40, 50, and 60) °C. The dose of charcoal was varied from  $0.13 \text{ g} \cdot \text{L}^{-1}$  to  $0.53 \text{ g} \cdot \text{L}^{-1}$  and mustard cake from  $2.0 \text{ g} \cdot \text{L}^{-1}$  to  $15 \text{ g} \cdot \text{L}^{-1}$  at pH 3.0, and the adsorbate concentration was  $2 \cdot 10^{-4} \text{ M}$ . The results are

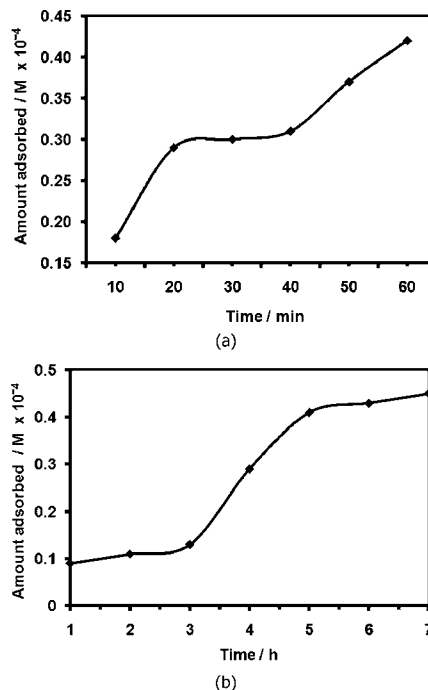


Figure 2. Plot of contact time vs adsorption of rhodamine-B over (a) charcoal and (b) mustard cake.

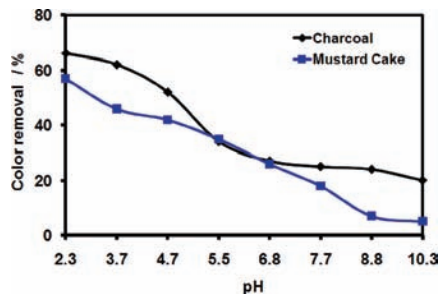


Figure 3. Plots of pH vs % color removal of rhodamine-B.

depicted in Figure 1. It was observed that for charcoal adsorption it increases from  $0.13 \text{ g} \cdot \text{L}^{-1}$  to  $0.40 \text{ g} \cdot \text{L}^{-1}$  and for mustard cake it increases from  $2 \text{ g} \cdot \text{L}^{-1}$  to  $12.5 \text{ g} \cdot \text{L}^{-1}$ . Thus adsorption increases with an increase in the dose of adsorbent due to availability of more binding sites for adsorption. However, a further increase in the dose of adsorbent did not affect the uptake capacity because of the unavailability of adsorbate sites due to saturation.

**3.3. Effect of the Contact Time.** The adsorption experiments were carried out at different contact times with a fixed adsorbent dose ( $0.26 \text{ g} \cdot \text{L}^{-1}$  of charcoal and  $5.0 \text{ g} \cdot \text{L}^{-1}$  of mustard cake), a temperature of 60 °C for charcoal and with mustard cake at 40 °C, pH 3.0, and adsorbate concentration of  $2 \cdot 10^{-4} \text{ M}$  (Figure 2). It is apparent from the figure that the optimum time is 60 min and 6 h for charcoal and mustard cake, respectively, which was sufficient for the maximum adsorption of dye.

**3.4. Effect of pH.** The influence of pH was studied over the pH range 2.30 to 10.30. pH profiles are shown in Figure 3. It is clear that adsorption decreases with an increase in pH. In case of charcoal the maximum uptake of the dye took place at pH 2.3 (66 % removal) and with mustard cake at pH 2.3 (57 % removal). Thus, this indicates that adsorption of the dye is influenced by the pH of the solution and the point of zero charge of the adsorbent ( $\text{pH}_{\text{zpc}}$ ). The point of zero charge of mustard cake ( $\text{pH}_{\text{zpc}}$ ) has been found to occur at pH 8.2. The adsorption of dye was higher at a solution pH <  $\text{pH}_{\text{zpc}}$ . This could be due

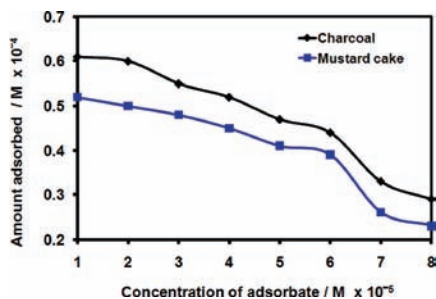


Figure 4. Plots of adsorbate concentration vs adsorption of rhodamine-B.

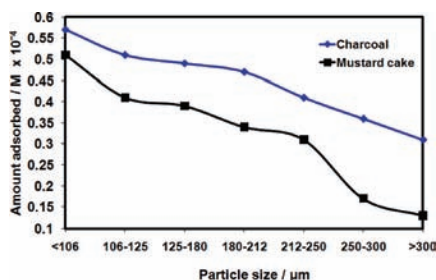


Figure 5. Plots of particle size of adsorbent vs adsorption of rhodamine-B.

to more positive charges at the mustard cake surface, resulting in the higher adsorption of the dye.

**3.5. Effect of Adsorbate Concentration.** For observing the effect of the concentration of the adsorbate, a dye concentration range of  $1.0 \cdot 10^{-5}$  M to  $8 \cdot 10^{-5}$  M was selected at temperatures of 60 °C for charcoal and 40 °C for mustard cake and contact times (60 min for charcoal and 6 h for mustard cake) as shown in Figure 4. Here, adsorption decreases with an increase in concentration of adsorbate due to a decrease in readily available vacant sites as the dye concentration is increased.

**3.6. Effect of Particle Size of Adsorbent.** The adsorption studies were carried out at seven different particle sizes which are (< 106, 106 to 125, 125 to 180, 180 to 212, 212 to 250, 250 to 300, and > 300)  $\mu\text{m}$  (Figure 5). It is observed that as the particle size decreases, the adsorption of the dye increases. Maximum adsorption with charcoal was 80 % and with mustard cake was 71 % at a mesh size of < 106  $\mu\text{m}$ . This is due to larger surface area associated with smaller particles.

**3.7. Effect of Temperature.** Adsorption studies were carried out at (40, 50, and 60) °C with both adsorbents as exhibited in Figure 6. It is apparent that the adsorption of rhodamine-B on charcoal increases with an increase in temperature, signifying the process to be endothermic in nature. However, with mustard cake the process is found to be exothermic in nature because of the expansion of the pores of the adsorbent on higher temperature which releases some of the dye molecules from the interior of the adsorbent. The result probably depends on the nature of the adsorbent used.

**3.8. COD Determination.** The chemical oxygen demand (COD) of the adsorbed dye solution gets considerably reduced after adsorption on charcoal and mustard cake. The COD of the solution after adsorption shows a significant decrease from  $1230.0 \text{ mg} \cdot \text{L}^{-1}$  to  $256.0 \text{ mg} \cdot \text{L}^{-1}$  for charcoal and from  $1230.0 \text{ mg} \cdot \text{L}^{-1}$  to  $258.0 \text{ mg} \cdot \text{L}^{-1}$  for mustard cake, indicating a lower toxicity level of the solution left after adsorption.

**3.9. Adsorption Isotherms.** Adsorption isotherms are useful in describing how solutes interrelate with the adsorbent and so

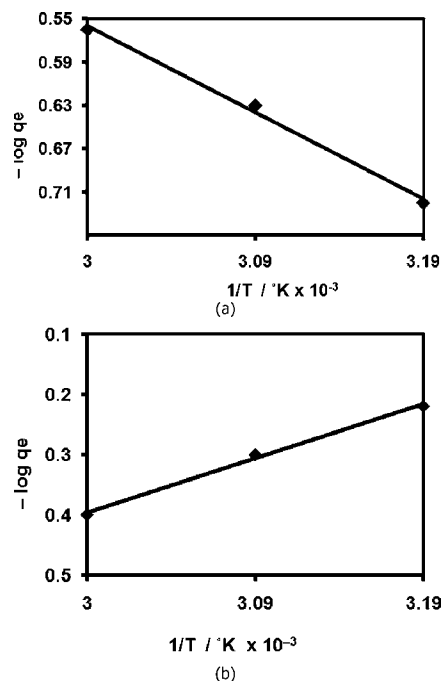


Figure 6. Plot of the effect of temperature on the adsorption of (a) rhodamine-B-charcoal and (b) rhodamine-B-mustard cake adsorption systems.

are critical in optimizing the use of adsorbents. The Langmuir and Freundlich isotherms were used to analyze the adsorption results.

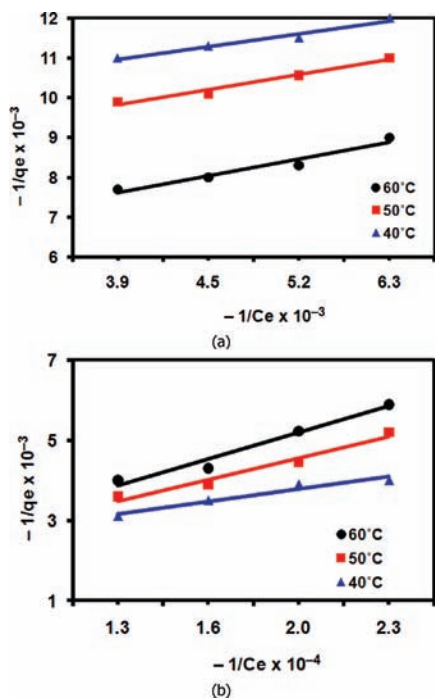
**3.9.1. Langmuir Isotherms.** Langmuir isotherms have been found to have a successful application in many real sorption processes.<sup>36</sup> The basic assumption is that, once a dye molecule occupies a site, no further sorption can take place at that site. The saturated monolayer curve can be represented by the equation,

$$\frac{1}{q_e} = \frac{1}{Q^\circ} + \frac{1}{bQ^\circ C_e} \quad (1)$$

where  $q_e$  is the amount adsorbed ( $\text{mol} \cdot \text{g}^{-1}$ ),  $C_e$  is the equilibrium concentration of the adsorbate ( $\text{mol} \cdot \text{g}^{-1}$ ), and  $Q^\circ$  and  $b$  are the Langmuir constants related to the maximum adsorption capacity and energy of adsorption, respectively.

When  $1/q_e$  was plotted against  $1/C_e$  (Figure 7), a straight line with slope  $1/bQ^\circ$  at temperatures of (40, 50, and 60) °C was obtained, indicating that the adsorption of the dye on charcoal and mustard cake follows the Langmuir isotherm. The values of the Langmuir constants  $b$  and  $Q^\circ$  are tabulated in Table 1.

The essential characteristics of isotherms can be expressed in terms of the dimensionless constant separation factor  $R_L$ , defined by Hall et al.<sup>37</sup> The parameter  $R_L$  indicates the isotherm to be irreversible ( $R_L = 0$ ), favorable ( $0 < R_L < 1$ ), linear ( $R_L = 1$ ), and unfavorable ( $R_L > 1$ ). The best-fitting isotherm is frequently determined by linear regression, and the applicability of isotherm equations is compared by statistically judging the coefficients of determination. The coefficients of determination reported in Table 1 showed strong positive evidence that the adsorption of rhodamine-B dye onto mustard cake follows the Langmuir isotherm. It is also clear from Table 1 that the  $R_L$  values are less than unity, thereby confirming a favorable adsorption process.



**Figure 7.** Langmuir adsorption isotherm for the (a) rhodamine-B–charcoal and (b) rhodamine-B–mustard cake adsorption systems at different temperatures.

**Table 1.** Langmuir Constants for the Adsorption of Rhodamine-B over Charcoal and Mustard Cake

$T$ °C	charcoal				mustard cake			
	$b$ L·mol <sup>-1</sup>	$Q^{\circ}$ mol·g <sup>-1</sup>	$R^2$	$R_L$	$b$ L·mol <sup>-1</sup>	$Q^{\circ}$ mol·g <sup>-1</sup>	$R^2$	$R_L$
40	0.4660	10.3	0.9927	0.943	0.482	2.9	0.9543	0.391
50	0.1298	7.7	0.9877	0.984	1.718	3.2	0.9635	0.225
60	0.5217	6.9	0.9922	0.937	0.777	3.6	0.9798	0.509

**3.9.2. Freundlich Isotherm.** The linear form of the Freundlich equation is represented as:

$$\log q_e = \log K_f + \frac{1}{n} \log c_e \quad (2)$$

where  $K_f$  and  $n$  are the Freundlich constants related to adsorption capacity and adsorption intensity, respectively.

The data obtained from the linear Freundlich isotherm plot (Figure 8) for the adsorption of the rhodamine-B dye onto mustard cake is presented in Table 2. The correlation coefficients ( $> 0.97$ ) showed that the process obeys the Freundlich isotherm as well. The calculated values of the Freundlich constants  $K_f$  and  $n$  are given in Table 2.

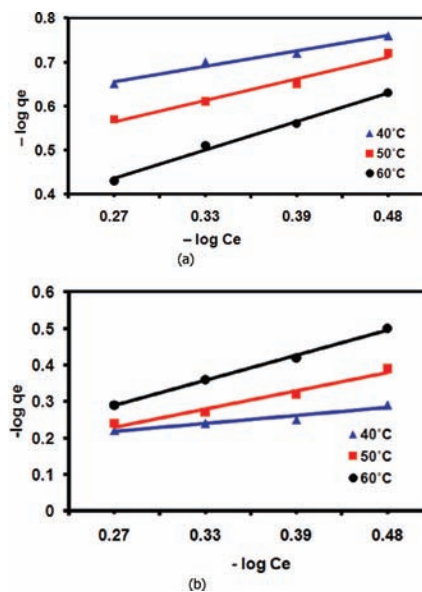
**3.10. Thermodynamic Parameters.** Thermodynamic parameters for the adsorption of rhodamine-B dye on charcoal and mustard cake were calculated using following equations, and their values are given in Table 3.

$$\Delta G^{\circ} = -RT \ln b' \quad (3)$$

$$\ln b_2/b_1 = (\Delta H^{\circ}/R)(T_2 - T_1/T_2T_1) \quad (4)$$

$$\Delta S^{\circ} = -(\Delta G^{\circ} - \Delta H^{\circ})/T \quad (5)$$

where  $\Delta G^{\circ}$  is the change in Gibbs energy,  $\Delta H^{\circ}$  is the change in the enthalpy, and  $\Delta S^{\circ}$  is the change in the entropy.  $b'$ ,  $b_1$ ,



**Figure 8.** Freundlich adsorption isotherm for the (a) rhodamine-B–charcoal (b) rhodamine-B–mustard cake adsorption systems at different temperatures.

**Table 2.** Freundlich Constants for the Adsorption of Rhodamine-B over Charcoal and Mustard Cake

$T$ °C	charcoal			mustard cake		
	$K_f$ mol·g <sup>-1</sup> ·L·mol <sup>-1/n</sup>	$n$	$R^2$	$K_f$ mol·g <sup>-1</sup> ·L·mol <sup>-1/n</sup>	$n$	$R^2$
40	2.05	0.777	0.9651	0.60	0.888	0.972
50	3.03	0.555	0.9982	12.30	0.200	0.9917
60	2.06	0.666	0.9818	9.79	0.333	0.9959

**Table 3.** Thermodynamic Parameters of Rhodamine-B with Charcoal and Mustard Cake

adsorbent	$\Delta G^{\circ}$ J·mol <sup>-1</sup>	$\Delta H^{\circ}$ kJ·mol <sup>-1</sup>	$\Delta S^{\circ}$ J·K <sup>-1</sup> ·mol <sup>-1</sup>
charcoal	-862.95	54.012	169.80
mustard cake	-824.80	-30.81	-92.82

and  $b_2$  are the Langmuir constants at (40, 50, and 60) °C, respectively.

The negative value of  $\Delta G^{\circ}$  indicates that the adsorption of rhodamine-B on both adsorbents is spontaneous. The positive value of  $\Delta H^{\circ}$  of rhodamine-B with charcoal confirms the endothermic nature of adsorption, whereas the negative value of  $\Delta H^{\circ}$  with mustard cake proves that the adsorption is exothermic in nature. The values of  $\Delta S^{\circ}$  reflect the affinity of adsorbents toward the dye.

**3.11. Desorption Studies.** After determining the sorption profile, the possibility of recycling the adsorbent was investigated. An adsorption column chromatography method<sup>38,39</sup> was adopted. A portion of 400 mg of mustard cake was packed in the column. The length of the packing in the column was set to 4.5 cm. Three different concentrations of ethanol, that is, 50 %, 40 %, and 30 %, were used as the eluting agent. The eluent was passed through the column at flow rate of 5 mL·min<sup>-1</sup>, and fractions collected after every 10 min, which were analyzed spectrophotometrically.

With 50 % ethanol, about 80 % of rhodamine-B was desorbed after 1 h; with 40 % ethanol, the desorption percentage decreased to 60 %, and with 30 % ethanol, only 40 % was desorbed.

#### 4. Conclusions

On the basis of results obtained, it is concluded that mustard cake was efficiently utilized as an adsorbent for the removal of the dye rhodamine-B, and its efficiency is comparable with that of activated carbon. Mustard cake is cheap and is easily available from local oil mills and can act as a replacement for charcoal. As a waste product, it provides a solution for solid-waste management and solves the problem of its disposal. Regarding the rhodamine-B, it can be recycled and reused in industrials. Therefore, the reported study is an environmentally friendly method. This study recommends that the mustard cake could be employed as a low-cost adsorbent as alternatives to commercial activated carbon for the removal of dyes from wastewater.

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