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Removal of Acid Violet 17 from aqueous solutions by adsorption onto activated carbon prepared from sunflower seed hull

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Abstract

The adsorption of Acid Violet 17 (AV17) was carried out using various activated carbons prepared from sunflower seed hull (SSH), an agricultural solid waste by-product. The effect of parameters such as agitation time, initial dye concentration, adsorbent dosage, pH and temperature were studied. The Langmuir and Freundlich isotherm models were applied and the Langmuir model was found to best report the equilibrium isotherm data. Langmuir adsorption capacity was found to be 116.27 mg/g. Kinetic data followed pseudo-second-order kinetics. Maximum colour removal was observed at pH 2.0. It was observed that the rate of adsorption improves with increasing temperature and the process is endothermic. The adsorbent surface was analysed with a scanning electron microscope. The results indicate that activated sunflower seed hull could be an attractive option for colour removal from dilute industrial effluents.

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Keywords: Sunflower seed hull; Acid Violet 17; Adsorption; Isotherms; Kinetics

1. Introduction

Acid dyes are used by several industries, such as textile, paper, printing and plastics to colour their products [1,2]. The effluent discharged from these industries is highly coloured and disposal of this coloured water into the receiving water body not only causes damage to aquatic life, but also to human beings, by producing carcinogenic and mutagenic effects [3,4]. There are several treatment technologies like photo degradation [5], biodegradation [6], coagulation flocculation [7] and electrochemical oxidation [8,9] available for the treatment of coloured wastewater. Among the numerous techniques of colour removal, adsorption [10] is considered to be one of the more efficient and less expensive methods. Most of the commercial industries use activated carbon as adsorbent to remove colour from wastewater [11]. However, its use is often limited due to its high cost. Attempts have been made to find alternative low-cost adsorbents [12]. Agricultural waste materials have little or no economic value and often pose a disposal problem. So, activated carbon

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prepared from these wastes help to solve the waste disposal problem. Agricultural wastes include coir pith [13], orange peel [14], Indian rosewood [15], jute fibre [16], wheat shells [17], soy meal hull [18], rice husk [19], activated date pit [20], bamboo dust [21], etc. In a developing country like India where agriculture is the primary occupation, agricultural waste by-product such as sunflower seed hulls are abundantly available. Therefore, it would be worthwhile to develop a low-cost adsorbent from sunflower seed hulls. In the present study, three different activated carbons prepared from sunflower seed hulls have been used as adsorbent for colour removal of Acid Violet 17 (AV17). The effects of operating parameters such as initial dye concentration, adsorbent dosage, solution pH, kinetics and temperature were studied.

2. Experimental

2.1. Materials and methods

Sunflower seed hulls were collected from a dehulling unit at Dindugul, Tamilnadu, India. The adsorbate AV17 was obtained from Hindustan Ciba-Geigy, Mumbai, India and was used as such without any purification. The structure of Acid Violet 17

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Fig. 1. Chemical structure of Acid Violet 17.

is shown in Fig. 1. Sulphuric acid used for the activation of sunflower seed hull, hydrochloric acid and sodium hydroxide used for the pH studies were of analytical reagent grade and were purchased from Qualigens Fine Chemicals, Mumbai, India.

2.2. Preparation of activated adsorbents

The raw material, sunflower seed hulls were repeatedly washed with distilled water to remove dirt, dust and other impurities. The washed hulls were then sun dried for 48 h and treated with 18N sulphuric acid at three different temperatures. The first sample (SSH1) was prepared by immersing sunflower seed hulls in 18N sulphuric acid, 1:2 (w/v) for 24 h at room temperature. The second sample (SSH2) was prepared by immersing sunflower seed hulls in 18N sulphuric acid, 1:2 (w/v) for 24 h at 353 K in a muffle furnace (Gambaks Instruments Co, Chennai, India). The third sample (SSH3) was prepared by immersing sunflower seed hulls in 18N sulphuric acid, 1:2 (w/v) for 24 h at 393 K in a muffle furnace. After treatment all the samples were cooled to room temperature, washed with distilled water until the filtrate reached neutral pH. The resulting activated carbon materials were dried in hot air oven at 105 °C, powdered and sieved through a 0.6 mm sieve.

2.3. Characterization of the adsorbents

The surface area of all samples was measured by N_2 adsorption at 77 K using Nova 4200e (Quantachrome Corp.). The BET surface area was obtained by applying BET equation to the adsorption data. The particle morphology of the prepared samples was examined using Philips XL 30 scanning electron microscope (SEM). The surface areas (BET) and SEM images of the adsorbents studied are given in Table 1 and Fig. 7, respectively.

2.4. Batch adsorption experiments

To study the effect of parameters like adsorbent dosage, pH and initial concentration for the colour removal of AV17, batch

Table 1				
BET surface area	of S	SH1,	SSH2,	SSH3

Adsorbent	$S_{\rm BET} (m^2/g)$
SSH1	10.35
SSH2	18.19
SSH3	21.06

experiments were carried out in a thermostatic orbital shaker (Orbitek, Scigenics Biotech Pvt. Ltd., Chennai, India) at a constant speed of 175 rpm at 303 ± 1 K. The adsorption isotherm experiment was carried out by agitating 50 mL of dye solution of various concentrations. After agitation, the dye solutions were separated from the adsorbent by centrifugation (Research Centrifuge, Remi Scientific Works, Mumbai, India) for 5 min. The dye removal was determined spectrophotometerically (Shimadsu model: UV 1601) by monitoring the absorbance changes at the wavelength of maximum absorbance (587 nm). The effect of pH on dye removal was studied over a pH range of 2-10. The pH of the dye solution was adjusted by the addition of dilute HCl or NaOH (0.1 M) solutions. For the optimum amount of adsorbent per unit mass of the adsorbate, a 50 mL dye solution was contacted with different amounts of various activated SSH, till equilibrium was reached. The kinetics studies were carried out using a mechanical stirrer. The samples were withdrawn at predetermined time intervals and centrifuged. The effect of temperature on the adsorption characteristics was studied by determining the adsorption isotherms at 308, 313 and 318 K at a fixed dye concentration and dosage. The dye removal percentage and amount adsorbed were calculated using the following relationships:

Percentage removal =
$$\frac{C_{\rm i} - C_{\rm f}}{C_{\rm i}} \times 100$$
 (1)

Amount adsorbed
$$(q_e) = \frac{(C_i - C_f)V}{m}$$
 (2)

where C_i and C_f are the initial and final concentrations (mg/L) of dye, respectively, *m* the mass (g) of adsorbent and *V* is the volume of dye solution (mL).

3. Results and discussion

3.1. Effect of adsorbent dosage

The effect of mass on the removal of AV17 by various activated SSH is shown in Fig. 2. This figure reveals that the removal of AV17 increases upto a certain limit and then it remains almost constant. An increase in adsorption with adsorbent dosage can be attributed to increased surface area and the availability of more adsorption sites [15]. But the amount adsorbed per unit mass of the adsorbent decreased considerably. The decrease in unit adsorption with increasing dose of adsorbent is basically due to the adsorption sites remain unsaturated during the adsorption process [22]. For the quantitative removal of dye from 50 mL of 120 mg/L a maximum dosage of 100 mg of adsorbent is required.



Fig. 2. Effect of adsorbent dosage on the adsorption of Acid Violet 17 onto SSH1, SSH2, SSH3. Conditions: t = 8 h, C_0 : 120 mg/L.

3.2. Effect of pH

The effect of pH on the adsorption of dye by various activated SSH was studied by varying the pH of the dye solution from 2.0 to 10 for an initial concentration of 100 mg/L (Fig. 3). Maximum adsorption occurs at acidic pH (pH 2). The lower adsorption of AV17 an anionic dye, at alkaline pH is because of the presence of excess OH⁻ ions competing with the dye anions for the adsorption sites. At pH 2, a significantly high electrostatic attraction exists between the positively charged surface of the adsorbent and negatively charged anionic dye [23]. As the pH of the system increases, the number of negatively charged sites increases and the number of positively charged sites decreases. A negatively charged surface site on the adsorbent did not favour the adsorption of anionic dye due to electrostatic repulsion. A similar result was observed for the adsorption of acid red 14 by soy meal hull [18] and Congo red by baggese fly ash [24].



Fig. 3. Effect of pH for the adsorption of Acid Violet 17 onto onto SSH1, SSH2, SSH3. Conditions: t = 8 h, C_0 : 100 mg/L, m: 0.1 g/50 mL.

3.3. Isotherm studies

The equilibrium adsorption isotherm is of importance in the design of adsorption systems. In general, the adsorption isotherm describes how adsorbates interact with adsorbents and this is critical in optimising the use of adsorbents. Two commonly used isotherms such as Langmuir and Freundlich were selected for this study. The Langmuir adsorption isotherm is based on the assumption [25] that adsorption takes place at specific homogeneous sites within the adsorption takes place at that site. Theoretically, the sorbent has a finite capacity to adsorb the sorbate. Therefore, a saturation value is reached beyond which no further sorption takes place [26]. The monolayer capacity can be represented by the expression:

$$q_{\rm e} = \frac{Q_{\rm m} K_{\rm L} C_{\rm e}}{1 + K_{\rm L} C_{\rm e}} \tag{3}$$

The linear form of the above equation is represented as:

$$\frac{C_{\rm e}}{q_{\rm e}} = \frac{1}{Q_{\rm m}K_{\rm L}} + \frac{C_{\rm e}}{Q_{\rm m}} \tag{4}$$

where C_e is the concentration of the dye solution (mg/L) at equilibrium, q_e the amount of dye adsorbed per unit weight of adsorbent (mg/g) and K_L is the constant related to the free energy of adsorption (L/mg). Q_m is the maximum adsorption capacity. The values of Q_m and K_L were calculated from the slope and intercept of the linear plot.

An essential characteristic of Langmuir isotherm can be expressed in terms of a dimentionless constant called equilibrium parameter [17]:

$$R_{\rm L} = \frac{1}{1 + K_{\rm L}} C_0 \tag{5}$$

where $K_{\rm L}$ is the Langmuir constant and C_0 is the highest initial dye concentration (mg/L). The value of $R_{\rm L}$ indicates the type of isotherm to be favourable ($0 < R_{\rm L} < 1$), linear ($R_{\rm L} = 1$), unfavourable ($R_{\rm L} > 1$) or irreversible $R_{\rm L} = 0$.

Freundlich isotherm is an emprical equation employed to describe heterogeneous systems. The Freundlich equation is commonly given by:

$$q_{\rm e} = K_{\rm F} C_{\rm e}^{1/n} \tag{6}$$

where q_e is the amount of solute adsorbed per unit weight of adsorbent (mg/g), C_e the equilibrium concentration of solute in the bulk solution (mg/L), K_F the Freundlich constant indicative of the relative adsorption capacity of the adsorbent (mg/g) and 1/n is the heterogeneity factor. A linear form of the Freundlich expression can be obtained by taking logarithms of the non-linear form:

$$\log q_{\rm e} = \log K_{\rm F} + \frac{1}{n} \log C_{\rm e} \tag{7}$$

A plot of $\log q_e$ versus $\log C_e$ enables the constant K_F and exponent 1/n to be determined from the intercept and slope of the line, respectively.



Fig. 4. Langmuir isotherm plots for adsorption of Acid Violet 17 onto SSH1, SSH2, SSH3. Conditions: t = 8 h, m: 0.05 g/50 mL.



Fig. 5. Freundlich isotherm plots for adsorption of Acid Violet 17 onto SSH1, SSH2, SSH3. Conditions: t = 8 h, m: 0.05 g/50 mL.

The adsorption isotherm data of AV17 onto various activated SSH were fitted with both Langmuir (Fig. 4) and Freundlich isotherms (Fig. 5). The correlation coefficient R_L^2 is comparatively higher than R_F^2 . Moreover, the dimentionless constant R_L lies within the favourable limit. Fig. 6 shows the comparitive fit of Langmuir and Freundlich isotherms with the equilibrium data plotted as q_e versus C_e . It is clear from the figure that the Langmuir isotherm shows good fit with the data. The results indicate that the Langmuir model is the best-fit model for the adsorption of AV17 onto various activated SSH. The calculated



Fig. 6. The fit of experimental adsorption data to Langmuir and Freundlich models for AV17 onto SSH1, SSH2, SSH3.

isotherm constants are given in Table 2. Among the adsorbents studied, SSH2 showed the highest adsorption capacity when compared to other adsorbents, SSH1 and SSH3. SSH1 had a low adsorption capacity because it was activated with 18N H₂SO₄ at room temperature, resulting in the formation of a less number of pores and a smaller surface area [20,27]. SSH3 activated with 18N H₂SO₄ at a temperature of 120 °C shows slight reduction in the adsorption capacity even though it has a large surface area. It is possible that some functional groups involved in dye removal were destroyed upon this activation and thus the adsorption capacity was slightly reduced [28]. Having in mind that only pores whose openings are larger than the molecular size of the adsorbate are accessible to the adsorbate, it is also possible that the pore sizes generated upon activation are smaller than the molecular size of AV17, thus leading to lower adsorption. From the SEM images (Fig. 7), it is clear that SSH2 has a considerable numbers of pores and there is a good possibility for dyes to be trapped and adsorbed into these pores. According to the results obtained, SSH2 could be employed as lowcost adsorbent and could be considered as an alternative to commercial activated carbons for the removal of colour.

3.4. Kinetic studies

Several kinetic models can be used to express the mechanism of solute sorption onto a sorbent. In this study, pseudo-first-order and pseudo-second-order kinetic models were tested.

Table 2

Freundlich and Langmuir coefficients for adsorption of the dye AV17 onto SSH1, SSH2, SSH3

Adsorbent	Langmuir constar	nts			Freundlich constants			
	$\overline{Q_{\rm m}~({\rm mg/g})}$	$K_{\rm L}$ (L/mg)	$R_{\rm L}$	R^2	$\overline{K_{\rm F}~({\rm mg/g})}$	n	R^2	
SSH1	65.78	0.0857	0.0885	0.9914	9.77	2.18	0.9694	
SSH2	116.27	0.0890	0.0856	0.9915	20.54	2.48	0.9873	
SSH3	107.52	0.1341	0.0585	0.9977	22.51	2.43	0.9837	



Fig. 7. SEM photographs of SSH1, SSH2, SSH3.

Pseudo-first-order equation of Lagergren based on solid capacity is given by:

$$\log(q_{\rm e} - q_t) = \log q_{\rm e} - \frac{K_1 t}{2.303} \tag{8}$$

where q_e is the amount of dye adsorbed at equilibrium (mg/g), q_t the amount of dye adsorbed (mg/g) at time t, K_1 the equilibrium rate constant of pseudo-first-order kinetics (min⁻¹) and t is the contact time (min). The values of adsorption rate constant (K_1) for AV17 adsorption on various activated SSH were determined from the plot of $\log(q_e - q_t)$ against t (Fig. 8). These values are given in Table 3. The experimental data deviated greatly from the linearity. This was evidenced by low q_e and low correlation values. Moreover, first-order equation fits well when concentration of the solute is low, but at high concentration it deviates. So, the first-order Lagergren model was inapplicable to this system. Similar results were observed earlier for the adsorption of Acid Red 57 [29].

A linear form of pseudo-second-order model [30] is given by:

$$\frac{t}{q_t} = \frac{1}{K_2 q_{\rm e}^2} + \frac{1}{q_{\rm e}} t \tag{9}$$

where K_2 is the equilibrium rate constant of the pseudo-secondorder adsorption (g/(mg min)). The values of K_2 and q_e were

Table 3

Calcu	lated	and	experimenta	l q _e va	lues and	l t	he pseuc	lo-i	first-ord	ler and	i pseuc	lo-secon	d-orc	ler ra	ıte	cons	stan	t
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Adsorbent	Intial concentration (C_0)	$q_{\rm e}$ (mg/g) Exp	K_1 (min ⁻¹)	$q_{\rm e}$ (mg/g) Cal	R^2	$K_2 (\times 10^{-3} \text{ g/(mg min)})$	$q_{\rm e}$ (mg/g) Cal	<i>R</i> ²
	60	19.36	0.0119	18.12	0.983	0.76	22.93	0.974
SSH1	80	28.94	0.0133	16.57	0.9814	1.72	30.39	0.9918
	100	34.19	0.017	10.2	0.9828	4.46	34.84	0.9993
	60	44.65	0.0149	23	0.9921	1.39	46.72	0.9977
SSH2	80	50.65	0.014	21.07	0.9711	1.80	52.35	0.9994
	100	66.54	0.0145	30.57	0.9927	1.18	68.49	0.9981
	60	30.32	0.0025	14.19	0.8108	2.11	32.36	0.9974
SSH3	80	41.35	0.0083	13.87	0.7489	3.30	42.91	0.9997
	100	52.37	0.0032	24.43	0.5845	3.80	54.05	0.9997



Fig. 8. Acid Violet 17 uptake by SSH1, SSH2, SSH3 according to pseudo-firstorder kinetic model. Conditions: V: 500 mL, m: 0.5 g.

calculated from the plot of t/q_t versus t (Fig. 9). The values of q_e , K_2 along with the correlation coefficients for the pseudo-firstorder and pseudo-second-order models are presented in Table 3. The $q_{e,exp}$ and the $q_{e,cal}$ values from the pseudo-second-order kinetic model are close to each other. The correlation coefficients are also closer to unity for pseudo-second-order kinetics than that for the pseudo-first-order kinetics. This suggests that the sorption system can be well represented by the pseudo-secondorder model for the adsorption of AV17 by various activated SSH.

3.5. Thermodynamic studies

The thermodynamic parameters such as change in standard free energy (ΔG°), enthalpy (ΔH°) and entropy (ΔS°) of adsorp-



Fig. 9. Acid Violet 17 uptake by SSH1, SSH2, SSH3 according to pseudosecond-order model. Conditions: V: 500 mL, m: 0.5 g.



Fig. 10. The plot of $\log K_d$ vs. 1/T for SSH1, SSH2, SSH3. Conditions: t = 8 h, C_0 : 100 mg/L, m: 1 g/L.

Table 4 Thermodynamic parameters calculated for the adsorption of AV17 onto SSH1, SSH2, SSH3

Adsorbent	ΔH° (kJ/mol)	ΔS° (kJ/mol)	ΔG° (kJ/mol) at 313 K
SSH1	123.288	0.39	-2.25
SSH2	152.449	0.50	-7.85
SSH3	103.892	0.34	-3.04

*C*₀: 100 mg/L, *m*: 1 g/L.

tion were determined using the following equations:

$$K = \frac{q_{\rm e}}{C_{\rm e}} \tag{10}$$

$$\Delta G^{\circ} = -RT \ln K \tag{11}$$

$$\log K = \frac{\Delta S^{\circ}}{2.303R} - \frac{\Delta H^{\circ}}{2.303RT}$$
(12)

where *R* is the gas constant, *K* the equilibrium constant and *T* is the solution temperature in Kelvin. ΔH° and ΔS° were calculated from the slope and intercept of the Van't Hoff plots of log *K* versus 1/T (Fig. 10). The results are given in Table 4. The negative values of ΔG° indicate the degree of spontaneity of the adsorption process. The positive values of ΔH° show that the adsorption is endothermic; the possible explanation for this beinge displacement of more than one water molecule by the AV17 ions for their adsorption process [31]. The positive value of ΔS° suggests increased randomness at the solid/solution interface during the adsorption of SSH towards AV17. Also the positive ΔS° value corresponds to an increase in the degree of freedom of the adsorbed species. Therefore, adsorption is favoured on AV17.

4. Conclusion

The removal of Acid Violet 17 with various activated sunflower seed hull was systematically investigated under various conditions. The adsorption isotherm was best defined by the Langmuir isotherm model and the adsorption capacity was found to be 65.8, 116 and 65.8 mg/g for SSH1, SSH2 and SSH3, respectively. The kinetic experimental results were fitted with the pseudo-second-order model. Acidic pH is more favourable for this adsorption process. The thermodynamic studies showed that the process was endothermic and spontaneous in nature. The high adsorption capacity of prepared SSH adsorbents showed that these lowcost adsorbent could be used as an alternate for colour removal of various anionic dyes.

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