Separation Science and Technology
Publication details, including instructions for authors and subscription information:
http://www.informaworld.com/smpp/title~content=t713708471

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To cite this Article Gerçel, Özgül and Gerçel, H. Ferdi(2009) 'Removal of Acid Dyes from Aqueous Solutions using Chemically Activated Carbon', Separation Science and Technology, 44: 9, 2078 — 2095

To link to this Article: DOI: 10.1080/01496390902885338

URL: http://dx.doi.org/10.1080/01496390902885338

Separation Science and Technology, 44: 2078–2095, 2009

ISSN: 0149-6395 print/1520-5754 online
DOI: 10.1080/01496390902885338

Removal of Acid Dyes from Aqueous Solutions using Chemically Activated Carbon

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Abstract: Textile dyes (Acid Yellow 17 and Acid Orange 7) were removed from its aqueous solution in batch and continuous packed bed adsorption systems by using thermally activated *Euphorbia macroclada* carbon with respect to contact time, initial dye concentration, and temperature. The activated carbon was prepared using a cheap plant-based material called *Euphorbia macroclada*, which was chemically modified with K_2CO_3. Lagergren-first-order and second-order kinetic models were used to fit the experimental data. Equilibrium isotherms were analyzed by Langmuir and Freundlich isotherms. Equilibrium data fitted well the Langmuir model in the studied temperature (25–55°C) ranges. The maximum adsorption capacity of AY17 and AO7 onto activated carbon was found to be 161.29 and 455 mgg^{-1}, respectively by Langmuir isotherm at 55°C. Breakthrough curves for column adsorption have also been studied. The desorption of dyes has been experimentally investigated using NaOH solution of pH 11.

Keywords: Acid dye, activated carbon, activation, adsorption, biomass

INTRODUCTION

Carbonaceous materials have long been known to provide adsorptive properties. Activated carbons are made by the thermal decomposition of various carbonaceous materials followed by an activation process. The raw materials for activated carbon include hard and soft woods,

Received 18 June 2008; accepted 6 February 2009.

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peat, coals, petroleum coke, bones, and nutshells, such as coconut. Two standard activation methods are used: gas and chemical. Gas activation first involves carbonization at 400–500°C to eliminate the bulk of the volatile matter, and then partial gasification at 800–1000°C to develop the porosity and surface area. Activation is carried out with gases such as carbon dioxide, flue gases, or steam. The other activation process that is also used commercially depends on the action of inorganic additives to degrade and dehydrate the cellulosic materials. This is called chemical activation, usually used for lignin-based precursors. Chemical activation can be carried out using, for example, zinc chloride (1,2), sulfuric acid (3), phosphoric acid (4,5) or potassium carbonate to produce an activated carbon directly from the raw material at temperatures between 500 to 900°C. Chemical activation of lignocellulosic materials is mainly directed toward the preparation of powdered activated carbon. However, if the objective is to prepare granular activated carbon the impregnation step has to be carried out with special care to ensure an intimate contact between the raw material and the activator reagent (6,7).

Recently, potassium salts such as K_2CO_3 have been used in order to prepare activated carbons with high specific surface area. The reason for the high specific surface area is thought to be related to the formation of atomic K by the following reactions:

$$2C + K_2CO_3 \longrightarrow 2K + 3CO \tag{1}$$

Thus atomic K may expand the interlayers of adjacent hexagonal network planes consisting of C atoms and enhance pore formation (8). In addition to Hayashi et al. (2002) (9) studied the production of activated carbon with K_2CO_3 and found that the carbon was consumed through the formation of CO above 1000 K and the specific surface area and the pore volume of activated carbon increased (10,11).

Various materials are used to produce activated carbon by chemical activation with K_2CO_3 and some of the most commonly used are wastes such as polyurethane foam (11), palm shell (12), almond shell, coconut shell, oil palm shell, pistachio shell, and walnut shell (9), phenol–formaldehyde, urea–formaldehyde (10), waste newspaper (13), corn cob (14), cork waste (15), lignin (16), and natural elutrilithe (17).

In the present study, an attempt was made to prepare activated carbon from *Euphorbia macroclada*. Using *E. macroclada*, which is a cheap plant-based material, powder activated carbon is manufactured by chemical activation with K_2CO_3. The genus *Euphorbia* belongs to the Euphorbiaceae, the sixth largest family among flowering plants. This genus alone accounts for one-sixth of the whole group of flowering plants, with about 1000 species ascribed to it. Species of the family

Euphorbiaceae are well known to be generally toxic and to show skin irritation (18,19). *Euphorbia macroclada* has been considered a suitable plant as a potential activated carbon source because it grows in large quantities in arid and sunny lands of the Anatolia (20).

MATERIAL AND METHODS

Chemicals

Buracid Orange II (C.I. Acid Orange 7 = AO7) was obtained from Burboya Bursa Dye and Chemical Industry Inc., Turkey and Acid Yellow 17 (AY17) from Sigma-Aldrich, Germany. The two anionic dyes studied are the practical grade and used as received. Double-distilled water was used throughout.

Preperation of Activated Carbon

Euphorbia macroclada (EM) were used as the starting material in the preparation of activated carbon. The samples of *EM* were collected from south-west Anatolia. Prior to use, the sample was air dried, ground with a high-speed rotary cutting mill, and then screened to give the fractions of 0.47 mm average particle size. During the activation procedure, *EM* was impregnated with aqueous solution of potassium carbonate (K_2CO_3: *EM* ratio 1:1, by weight) for 24 h and then filtered and the resulting chemical loaded *EM* were placed in a furnace and heated ($10°C\ min^{-1}$) to the final carbonization temperature of 850°C for 2 h in N_2 atmosphere (flow rate = $100\ mL\ min^{-1}$). After cooling, the activated carbon was repeatedly washed with deionized hot water and dried at 110°C. The carbonized material was sieved to 125 μm size and used for adsorption experiments.

Batch Equilibrium Studies

Anionic dye solutions were prepared by dissolving dye in distilled water to the required concentrations. The pH of dye solutions was adjusted to 2 with adding a small amount of dilute HCI or NaOH solution using pH meter. In experiments of equilibrium adsorption isotherm, the mixture of activated carbon (0.1 g and 0.05 g for AY17 and AO7, respectively), dye solution (50 mL) were stirred using a magnetic stirrer for 6 hours using a bath to control the temperature at constant temperatures of 25, 35, and 55°C. The concentration of the dye in the solution after equilibrium adsorption was determined spectrophotometrically by spectrophotometer (Shimadzu UV-2101PC) at λ_{max} of 404 and 484 nm for AY17 and AO7, respectively. Eq. (2) was used to calculate the amount of adsorption at equilibrium q_e ($mg\,g^{-1}$)

$$q_e = (C_0 - C_e)V/m \tag{2}$$

where C_0 and C_e are the initial and equilibrium solution concentrations ($mg\,dm^{-3}$), respectively. V is volume of the solutions (dm^3), and m is the weight of activated carbon (g) used.

Batch Kinetic Studies

Kinetic studies of adsorption were also performed at three concentrations of the adsorbates and adsorption was investigated as a function of time. In experiments of batch kinetic adsorption, a mixture of activated carbon (0.3 g and 0.15 for AY17 and AO7, respectively) and 150 mL dye solution were stirred using a magnetic stirrer with a water bath to control temperature. The pH was used throughout kinetic studies. The pH of this solution was adjusted to pH 2 with dilute HCI solution.

Column Studies

Column studies were conducted using a downflow technique and were performed in a fixed bed mini glass column with an inside diameter of 1.00 cm, a bed depth of 11.0 cm. For a liquid flow rate which is steady, a glass column was packed with glass bead (diameter of 3 mm). Carbon particles were wetted for 24 h in distilled water. This was then fed into the test column (which was also filled with water) as slurry and then allowed to settle for an additional 24 h. The dye solutions at initial concentration (60 mg dm^{-3} and 100 mg dm^{-3} for AY17 and AO7, respectively) were passed continuously through carbon. Separate columns were prepared by taking appropriate amounts of activated carbon and through the prepared columns, dye solutions was allowed to flow at the rate of 0.6 ml/min. The flow rate was controlled with a peristaltic pump. The column studies were performed at a room temperature. The breakthrough curve was obtained by plotting the ratio of effluent concentration to influent concentration against volume of collected volume, V.

Dye Recovery

Recovery of the adsorbed dyes was by eluting with NaOH solution of pH 11 through the exhausted columns at a flow rate of 0.6 mL min^{-1}. Alkaline NaOH solution was particularly selected for the desorption of acid dyes (AY17 and AO7). Acid dyes give the best desorption results with alkaline solvent of higher pH.

RESULTS AND DISCUSSION

Characterization of Activated Carbon

Nitrogen adsorption is a standard procedure for the determination of porosity of carbonaceous adsorbents. The nitrogen adsorption-desorption isotherms of activated carbon, used for calculating their Brunauer-Emmett-Teller (BET) surface areas and total pore volumes, were obtained by using a Quantachrome Inst. Autosorb1 analyzer. Figure 1 shows typical N_2 adsorption–desorption isotherms of the activated carbons obtained through the potassium carbonate activation of *EM* at 77 K. Prior to measuring the adsorption isotherm, the activated carbon was degassed at 300°C for 12 h. It was found that the adsorption isotherm is classified as type I (Langmuir isotherm) character. The development of micropores and mesopores can be clearly confirmed by the shape of the isotherms. This suggests that the activated carbon made from *EM* would be more microporous with a narrow pore size distribution and a hysteresis loop of isotherm shows the presence of mesopores (21). The specific surface area and total pore volume of activated carbon were 1048 m^2 g^{-1} and 0.614 mL g^{-1}, respectively. Pore volume distribution of activated carbon

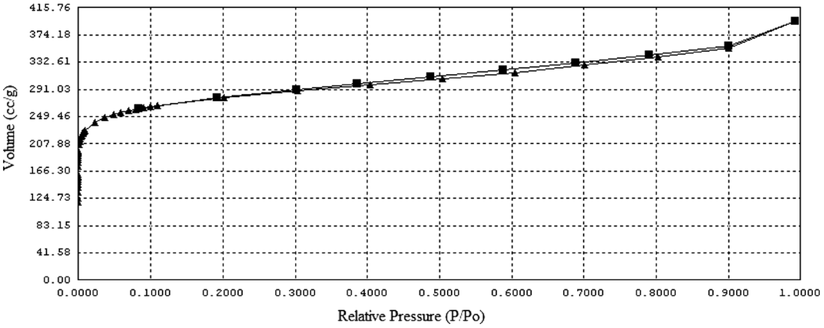

Figure 1. Adsorption-desorption isotherm of nitrogen at 77 K for activated carbon prepared from *E.M*, ▲ symbols, adsorption; ■ symbols, desorption.

was obtained using DFT model based on nitrogen adsorption and was shown in Fig. 2. The pores of adsorbents are generally classified into three groups, micropore (pore size <20 Å), mesopore (20–500 Å), and macropore (>500 Å). The pore size distribution curve indicates that most of the activated carbon consists of micropores (pore diameter less than 20 Å) (22).

Effect of Contact Time and Initial Dye Concentration

Figure 3 shows that the effect of initial dye concentration and contact time on the adsorption kinetics of the activated carbon at 25°C with respect to the anionic dyes (AY17 and AO7). An increase in initial dye concentration leads to an increase in the adsorption capacity of the dye on activated carbon and the adsorption increased with an increase in contact time. For the given concentrations the amounts of AY17 and AO7 adsorbed by activated carbon increased quickly with time in the beginning, then at a slower rate, and finally attained saturation called the equlibrium time, was dependent on time and initial concentration. A larger amount of dyes (for 120 mg dm^{-3} initial AY17 concentration and 400 mg dm^{-3} initial AO7 concentration) was adsorbed by activated carbon in the first 50 min of contact time and equilibrium was established about 60 and 90 min, respectively (23).

In order to investigate the mechanism of adsorption, the pseudo-first-order and the pseudo second-order adsorption models have been used in this study to predict adsorption kinetics. The first-order rate expression of Lagergren is given as (24,25):

$$\log(q_e - q_1) = \log q_e - k_1 t/2.303 \tag{2}$$

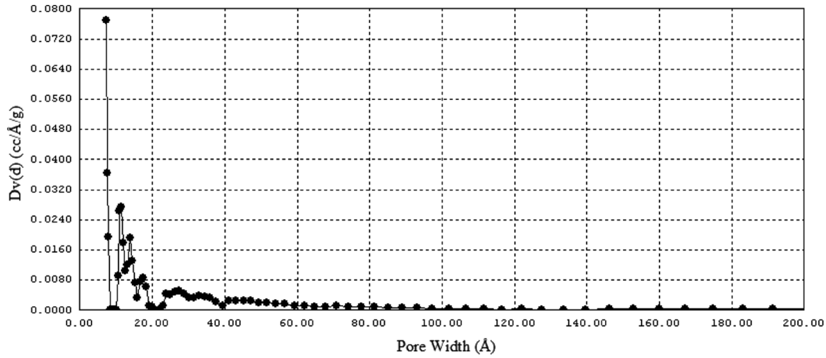

Figure 2. DFT/Monte-Carlo pore volume distribution of the carbons prepared from *EM*.

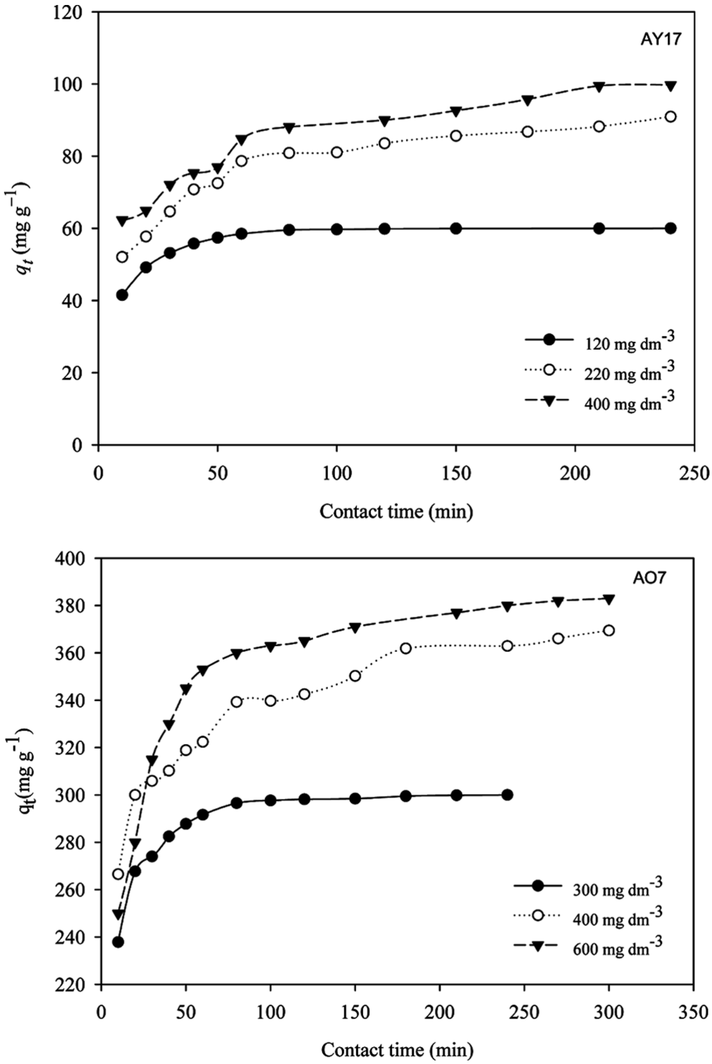

Figure 3. Effect of contact time for adsorption of AY17 and AO7 onto activated carbon at various initial dye concentrations.

where q_e and q_t are the amounts of dye adsorbed on adsorbent at equilibrium and at time t, respectively (mg g^{-1}) and k_1 is the rate constant of first-order rate constant (min^{-1}), was applied to the adsorption of acid dyes onto activated carbon. The slopes and intercepts of plots of log(q_e–q_t) vs. t were used to determine values of k_1. The pseudo-second-order kinetic model (26,27) is expressed as:

$$t/q_1 = 1/k_2 q_2^2 + (1/q_2)t \tag{3}$$

where q_2 is the maximum adsorption capacity (mg g^{-1}) for the pseudo-second-order adsorption, k_2 is the equilibrium rate constant for the pseudo-second-order adsorption (g mg^{-1} min^{-1}). Values of k_2 and q_2 were calculated from the plot of t/q_t against t.

The kinetic data for the adsorption of acid dyes (AY17 and AO7) at various initial dye concentrations were calculated from the related plots and were given in Table 1. The correlation coefficients for the pseudo-second-order kinetic model were higher than that of the pseudo-first-order. An increase in the initial concentration of dyes also leads to increase in the calculated q_2 values. These results imply that the adsorption system studied obeys to the pseudo-second-order kinetic model.

Effect of Temperature

It has been believed that the temperature generally has a major effect on the adsorption process. Changing the temperature will change the equilibrium capacity of the adsorbent for a particular adsorbate. The isotherms for adsorption of acid dyes (AY17 and AO7) onto activated carbon at three different temperatures are shown in Fig. 4. It was observed that the dye adsorption is highly dependent on the temperature of the solution. The equilibrium adsorption capacities of two acid dyes onto activated carbon were increased with increasing temperature from 25 to 55°C, thereby indicating the process to be endothermic and this effect may be due to the fact that at higher temperatures an increase in active sites occurs due to bond rupture. This could also be due to the improved mobility of dyes from the bulk solution towards the adsorbent surface (28).

The uptake of AO7 and AY17 reached a plateau showing the saturation of binding sites at higher concentration levels. It can be noticed that after saturation of binding sites,almost no more dye was adsorbed for all the temperatures suggesting that available sites on the activated carbon are the limiting factor for the adsorption (23). Dye properties are here the important factor determining the adsorption capacity, since the adsorbents are the same in each case. So, differences in acid dyes adsorption results was attributed to the different chemical natures of the two dyes, with respect to substituents and/or branched side chains that increase bulkiness and reduce diffusion ability (29). Furthermore, dye

Table 1. Kinetic parameteres for the adsorption of acidic dyes onto activated carbon at 55°C

Initial dye conc. ($mg\,dm^{-3}$)	Acid Yellow 17						Initial dye conc. ($mg\,dm^{-3}$)	Acid Orange 7					
	pseudo-first order			pseudo-second order				pseudo-first order			pseudo-second order		
	k_1	q_1	r^2	k_2	q_2	r^2		k_1	q_1	r^2	k_2	q_2	r^2
120	$2{,}63 \times 10^{-2}$	1,23	0,836	$4{,}31 \times 10^{-2}$	$6{,}02 \times 10^{+1}$	0,999	400	$3{,}45 \times 10^{-2}$	6,52	0,975	$1{,}08 \times 10^{-3}$	$4{,}00 \times 10^{+2}$	0,998
220	$7{,}23 \times 10^{-2}$	5,75	0,996	$2{,}04 \times 10^{-3}$	$1{,}15 \times 10^{+2}$	0,999	500	$2{,}99 \times 10^{-2}$	6,92	0,971	$7{,}45 \times 10^{-4}$	$4{,}55 \times 10^{+2}$	0,999
320	$1{,}75 \times 10^{-2}$	6,18	0,944	$6{,}33 \times 10^{-4}$	$1{,}52 \times 10^{+2}$	0,999	600	$4{,}58 \times 10^{-2}$	8,47	0,902	$7{,}47 \times 10^{-4}$	$4{,}76 \times 10^{+2}$	0,999

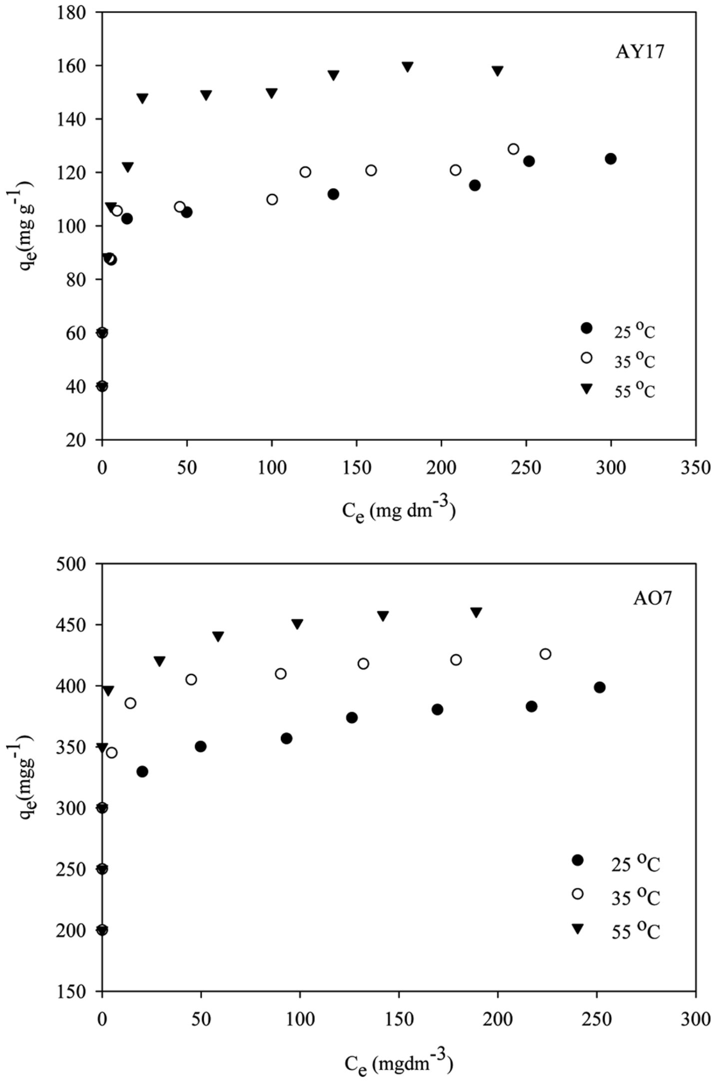

Figure 4. The isotherms for adsorption of acid dyes (AY17 and AO7) onto activated carbon at three different temperatures.

adsorption can be correlated with the sizes of dye molecules as well as the pore size distribution of activated carbon used. Unfortunately, the exact sizes of all dye molecules are not available.

The data for the adsorption of dyes (AY17 and AO7) onto activated carbon were fitted to Freundlich and Langmuir isotherms. The linear forms of the Langmuir and Freundlich isotherms equations (30,32) are represented by the following equation:

$$C_e/q_e = 1/q_{max}K_L + C_e/q_{max} \tag{4}$$

$$\text{In } q_e = \text{In } K_F + 1/n \text{ In} C_e \tag{5}$$

where, q_e is equilibrium dye concentration on the adsorbent (mg g^{-1}), C_e the equilibrium dye concentration in the solution (mg dm^{-3}), q_{max} the monolayer adsorption capacity of the adsorbent (mg g^{-1}), K_L is the Langmuir adsorption constant, and K_F (dm^3 g^{-1}) and n are Freundlich adsorption constants. The plots of C_e/q_e vs. C_e give a straight line of slope ($1/q_{max}$) and intercept ($1/q_{max}$ K_L) (Fig. 5). K_F and n can be determined from the linear plot of $\ln(q_e)$ vs $\ln(C_e)$ (not shown).

The Langmuir and Freundlich isotherm parameters for the adsorption of dyes (AY17 and AO7) onto activated carbon are listed in Table 2. It is evident from these results that the Langmuir isotherm model fits better than the Freundlich isotherm model, when the r^2 values of the models are compared in Table 2. One of the Langmuir constants q_{max} indicates the monolayer adsorption capacity of the adsorbent. The values of q_{max} increases, the monolayer adsorption capacity of adsorbent also increases, when the temperature increases 25–55°C for using acid dyes. The Langmuir constant q_{max} and K_L were influenced by temperature for both AY17 and AO7. For a AY17, increase in temperature from 25 to 55°C results in an increase in both q_{max} (from 125.00 to 161.29 mg g^{-1}) and K_L (from 0.137 to 0.281) indicating increasing the free energy of adsorption which is in confirmation with Langmuir isotherm constants. The maximum adsorption of AY17 and AO7 onto activated carbon was found to be 161.29 and 455 mg g^{-1}, respectively at 55°C.

The equilibrium adsorption capacity of acid dyes (AY17 and AO7) onto activated carbon was also affected by the temperature and increased with increasing temperature from 25 to 55°C which indicates that the adsorption of acid dyes (AY17 and AO7) onto activated carbon surface was favored at higher temperature and it is controlled by an endothermic process.

One of the essential features of the Langmuir isotherm could be expressed by a dimensionless constant called the separation factor or the equilibrium parameter, R_L,

$$R_L = 1/(1 + K_L C_0) \tag{6}$$

where C_0 is the initial maximum dye concentration. The values of R_L indicate the type of isotherm to be reversible ($R_L = 0$), favorable

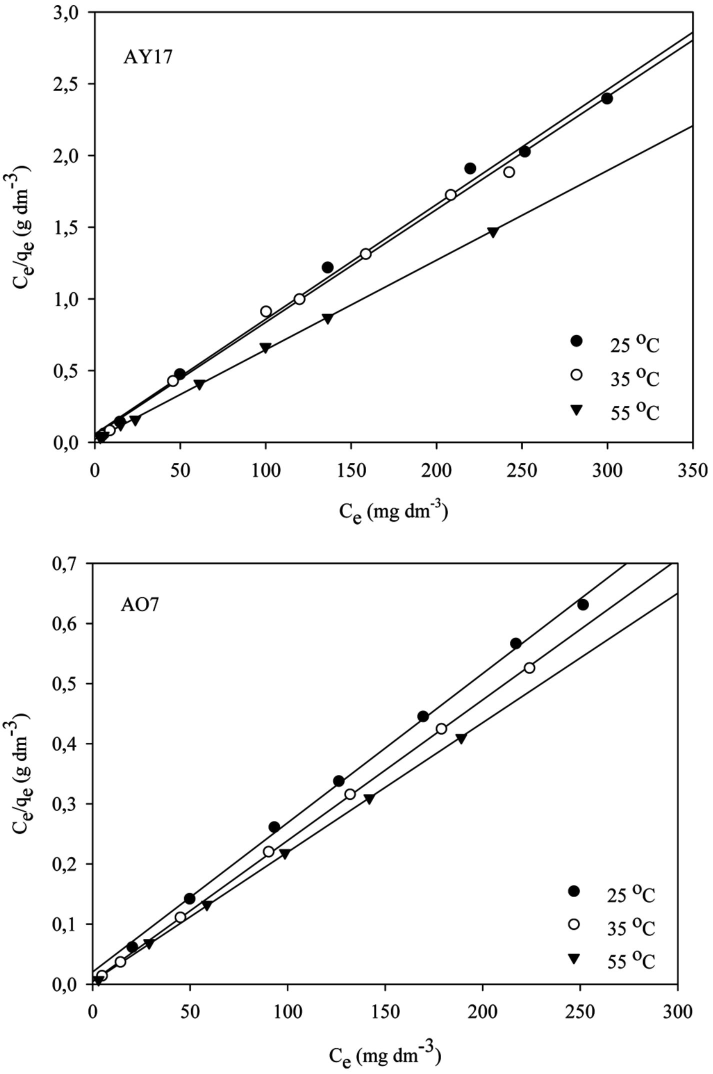

Figure 5. Langmuir plots for the adsorption of acid dyes (AY17 and AO7) onto activated carbon at three different temperatures.

($0 < R_L < 1$), linear ($R_L = 1$) or unfavorable ($R_L > 1$) (29). The values of R_L calculated as above equation are incorporated in Table 2. The R_L values of the adsorption of AY17 and AO7 onto activated carbon are between 0 and 1, therefore, their adsorption are favorable.

Table 2. Adsorption isotherm constants for the adsorption of acidic dyes onto activated carbon at various temperatures

Temp. (°C)	Acid Yellow 17							Acid Orange 7						
	Langmuir				Freudlich			Langmuir				Freudlich		
	q_{max}	K_L	r^2	R_L	$1/n$	K_F	r^2	q_{max}	K_L	r^2	R_L	$1/n$	K_F	r^2
25	$1{,}25 \times 10^2$	$1{,}37 \times 10^{-1}$	0,995	$1{,}3 \times 10^{-2}$	$7{,}7 \times 10^{-2}$	$7{,}90 \times 10^1$	0,918	$4{,}00 \times 10^2$	$1{,}19 \times 10^{-1}$	0,998	$1{,}27 \times 10^{-2}$	$7{,}0 \times 10^{-2}$	$2{,}65 \times 10^2$	0,958
35	$1{,}27 \times 10^2$	$1{,}58 \times 10^{-1}$	0,995	$1{,}1 \times 10^{-2}$	$7{,}3 \times 10^{-2}$	$8{,}23 \times 10^1$	0,850	$4{,}35 \times 10^2$	$4{,}50 \times 10^{-1}$	0,999	$3{,}39 \times 10^{-3}$	$4{,}9 \times 10^{-2}$	$3{,}27 \times 10^2$	0,931
55	$1{,}61 \times 10^2$	$2{,}81 \times 10^{-1}$	0,999	$6{,}0 \times 10^{-3}$	$1{,}26 \times 10^{-1}$	$8{,}48 \times 10^1$	0,863	$4{,}55 \times 10^2$	$4{,}68 \times 10^{-1}$	0,996	$3{,}27 \times 10^{-3}$	$3{,}8 \times 10^{-2}$	$3{,}76 \times 10^2$	0,970

Column Studies

Figure 6 shows the breakthrough curves for acid dyes (AY17 and AO7) adsorption from aqueous solutions onto the activated carbon in a fixed bed column. For both the columns, the inlet flow rate was kept at 0.8 mL min^{-1}. For a given initial dye concentration, the adsorption of AO7 onto activated carbon performed more efficiently. Thus, for an AO7 concentration of 100 mg dm^{-3}, the breakthrough volume 0.8 dm^3 for the column packed with activated carbon as against only 0.10 dm^3 using AY17 concentration of 60 mg dm^{-3}. In the present study, it may also be observed that the column was used for the adsorption of AY17 got fully exhausted within a shorter time when compared to the adsorption of AO7.

Desorption studies help in the recovery of the adsorbate and the adsorbent. Efficient elution of adsorbed solute from activated carbon in fixed-bed column is essential to ensure the reuse of activated carbon. NaOH solution of pH 11 has been used to elute AY17 and AO7 from activated carbon. In this study, NaOH solution was chosen for the desorption of acid dyes from the activated carbon as acid dyes give the best desorption results with alkaline solvent of higher pH. Desorption curves, the plots of effluent concentration (mg dm^{-3}) versus volume (dm^3) for AY17 and AO7 from the column, were given in Fig. 7. As shown in

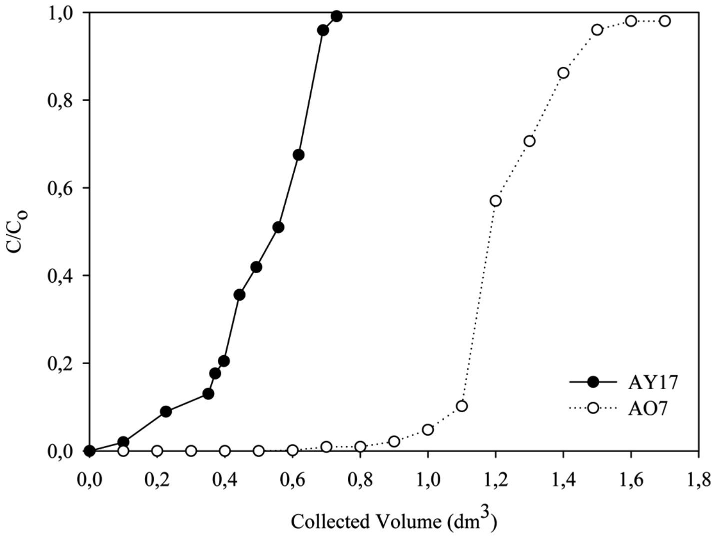

Figure 6. The breakthrough curves for acid dyes (AY17 and AO7) adsorption from aqueous solutions onto the activated carbon in a fixed bed column.

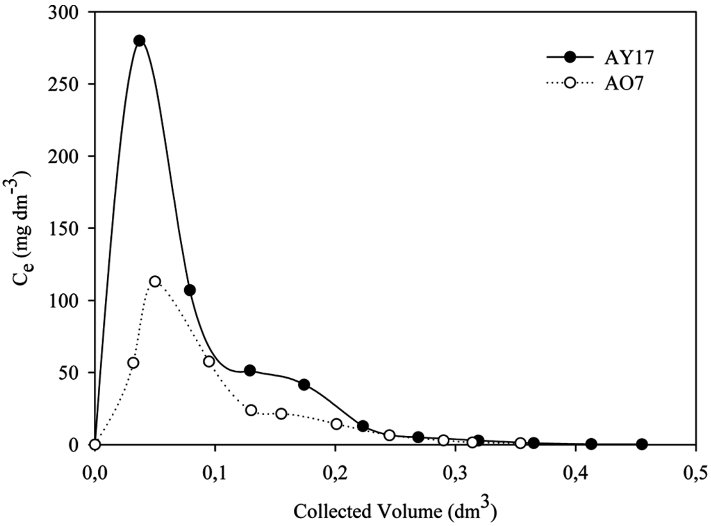

Figure 7. Desorption curves for AY17 and AO7 from a fixed bed column.

Figure 7, it is clear that the desorption efficiency of AY17 from activated carbon is apparently higher than the desorption efficiency of AO7. Adsorbed AY17 dyes could be readily desorbed from the activated carbon in the column. The majority of the adsorbed solute was desorbed in less than 200 mL for AY17. In contrast, the results showed that there is very little desorption of AO7 with alkaline solution from activated carbon.

CONCLUSIONS

In this study, the following results have been obtained:

- This study has indicated that high surface area activated carbons can be prepared from the chemical activation of *EM* with K_2CO_3 as activating agent and activated carbon prepared from *EM* may be used as an effective adsorbent material for the treatment of acid dye from aqueous solutions.
- In the study of kinetics of adsorption, the pseudo-second-order model provides better correlation of the adsorption data than the pseudo-first-order model.
- The Langmuir and Freundlich models also fit the isotherm data well, but the Langmuir model gives better fitting than Freundlich model.

- The equilibrium adsorption capacities of two acid dyes onto activated carbon were increased with increasing temperature from 25 to 55°C, thereby indicating the process to be endothermic.
- Adsorbed AY17 dyes could be readily desorbed from the activated carbon in the column. In contrast, the results showed that there is very little desorption of AO7 with alkaline solution from activated carbon.

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