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Acid Dyes Removal from Textile Wastewater Using Waste Cotton Activated Carbon: Kinetic, Isotherm, and thermodynamic studies

R. Salehi, F. Dadashian*, E. Ekrami
Department of Textile Engineering, Amirkabir University of Technology, P.O. Box: 15875-4413, Tehran, Iran.

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ABSTRACT

The present study aims at investigating the potential of activated carbon (AC) prepared from waste cotton fiber for the removal of Acid Dyes from aqueous solutions. The prepared activated carbon was characterized by pore structure analysis, Fourier transform infrared spectroscopy (FT-IR). Batch adsorption studies were carried out and the effect of experimental parameters such as pH, initial dye concentration and contact time on the adsorption was studied. The results revealed that the produced activated carbon has developed porous structure, fibrous shape, predominantly amorphous structure. The kinetic data obtained at different concentrations were investigated using the pseudo-first-order, pseudo-second-order and intraparticle diffusion models. The experimental data were found to conform to the pseudo-second-order kinetics with good correlation. The obtained equilibrium data were best fitted to the Freundlich model. Finally, according to the obtained thermodynamic parameters, it was found that the adsorption process was spontaneous and exothermic in nature. Prog. Color Colorants Coat. 11 (2018), 9-20© Institute for Color Science and Technology.

1. Introduction

Synthetic dyes are considered as a main class of organics which are mostly found in the environment due to their wide use in different industries. The textile industry uses a considerable volume of water and dyes for coloring its products. It has been estimated that more than 100,000 dyes are commercially available, however, the exact number and amount of them is not known. Most of the dyes have been found to be toxic or carcinogenic [1]. Because of the complexity of aromatic structure as well as the synthetic origin of these pollutants, their decolorization is usually difficult. Therefore, removal of dyes from textile effluents is one of the most important environmental concerns. Among several chemical and physical methods, the adsorption process is one of the effective techniques that have been successfully employed for dye removal from wastewater [2].

Adsorption phenomenon occurs as a result of the unsaturated and unbalanced molecular forces exist on any solid surface. So, as such surfaces come into contact with liquid or gas, an interaction occurs between the fields of forces of the surface and those of the liquid or gas. The solid surface to satisfy the residual forces by attracting and retaining the molecules, atoms or ions of the gas or liquid on its surface [3].

Activated carbon is the most widely used adsorbent,

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*Corresponding author: dadashia@aut.ac.ir

which is highly porous with a large surface area. However, its usage is very limited because of its expensiveness resulted from high production costs. Recently, interest in producing low cost activated carbon from industrial and agricultural solid wastes have been grown [4]. Activated carbon fibers (ACFs) are type of activated carbon which has gain considerable attractions due to their advantages over the conventional ones [5-10]. Powder and granule activated carbons suffer from lack of structure and flexibility, making them difficult to be fabricated without attaching to a web of polymer or other materials [11]. ACFs are found to produce more rapid dynamic adsorption characteristics in comparison to the granulated or powdered one, because of the small diameter of fibers resulting in the larger external surface area and minimized diffusion limitation [5, 8, 9, 12]. In this paper, the ability of waste cotton fiber based activated carbon as an adsorbent to remove acid dyes from aqueous solution is studied. The isotherm, kinetics and thermodynamics of dye adsorption is also investigated.

2. Experimental

2.1. Materials and Methods

Activated carbon was prepared using waste cotton fibers. Acid Red 94 and Acid Blue 45 were purchased from Merck. The chemical structures of Acid Red 94 and Acid Blue 45 are shown in Figure 1. The Activated carbon, produced under optimized conditions, was characterized by Fourier transform infrared spectroscopy. The surface area and pore volume of carbon nanostructures were characterized by BET nitrogen adsorption isotherm at 77 ºK. The pore size distribution was calculated from the desorption branch according to BJH method. The maximum wavelength used for determination of the residual concentration of Acid Red 94 and Acid Blue 45 using UV–vis spectrophotometer (Shimadzu, Model UV 1601, Japan) were 549 and 595 *nm*, respectively. The isotherm, kinetics and thermodynamics of dye adsorption were studied.

3. Result and Discussion

3.1. Characterization of activated carbon

Surface properties of activated carbon were investigated by studying FT-IR in the range of 500–4000 cm^{-1}. As shown in Figure 2, a strong and broad adsorption peak was observed at 3436 cm^{-1} which corresponds to the stretching of the OH functional group. It is also shown that bonded hydroxide there exists in the activated carbon sample. Activated carbon peaks at 2924 and 2854 cm^{-1} show the asymmetric and symmetric CH bonds, respectively.

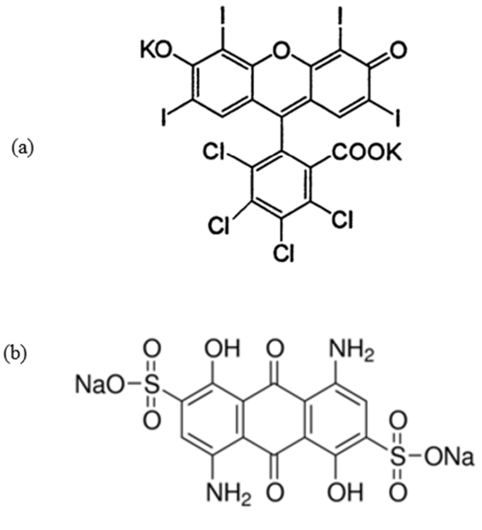


Figure1: Chemical structure of (a) Acid Red 94 and (b) Acid Blue 45.

These two peaks together with the peak at 1458 cm^{-1} are corresponding to the alkyl groups including methyl and methylene groups. The C=O stretching was observed because of carboxylic groups while the aromatic skeletal vibration was by the band centred at 1560 cm^{-1}. The broad band between 900 and 1300 cm^{-1} has a maximum at 1088 cm^{-1}, which can be attributed to P–O symmetrical vibration in a chain of P–O–P (polyphosphate) [13]. In general, phosphorus and phosphor carbonaceous compounds are characterized by absorption in this region [4].

Pore size distributions (PSD) of AC prepared at optimized conditions using BJH method is shown in Figure 3. PSD provides some information on the fraction of the total pore volume accessible to adsorbate molecules of a given size and shape. The activated carbon contains a certain amount of mesopores that is necessary to provide transport channels for adsorbate adsorption [11]. Adsorbents pores are classified into micropores (d<2 nm), mesopores (d=2-50 nm) and macropores (d>50 nm), as provided in IUPAC classification. The BJH plot for the N_2 adsorption/ desorption isotherm of the produced activated carbon represented microporosity, in which the majority of the pore sizes are in the range between 1.5 and 3 nm with an average pore diameter of 1.9 nm. The distribution of pore volume decreases significantly with the size of the pores, becoming insignificant in the macropores region. Total micropore (V_{micro}) and mesopore volumes (V_{meso}) were 0.2886 mL g^{-1} and 0.0670 mL g^{-1}, respectively.

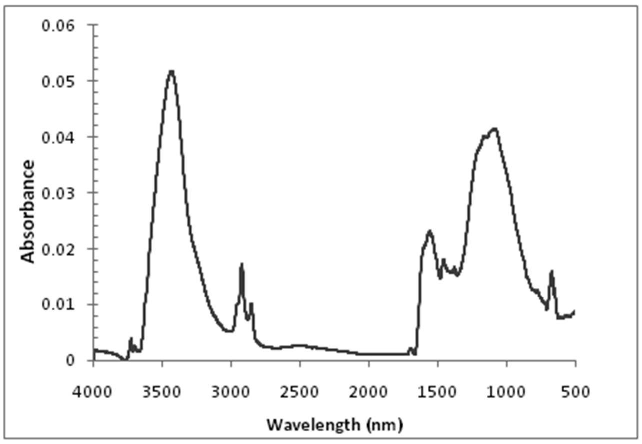


Figure 2: The FT-IR spectrum of the WCAC.

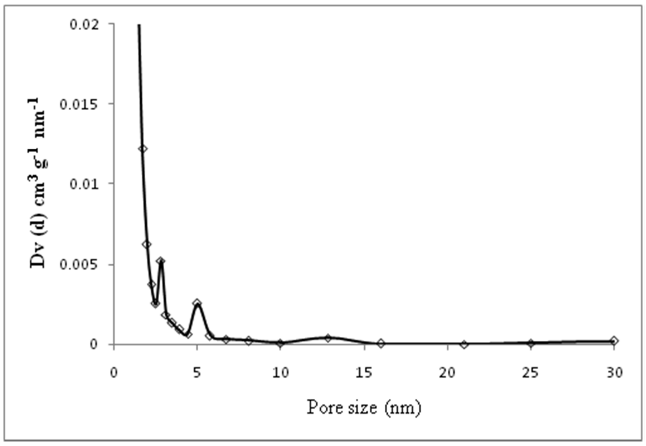


Figure 3: Pore size distributions of the ACF prepared at optimized conditions by BJH method.

3.2. Effect of operational parameters on dye removal

3.2.1. Solution pH effect

Figure 4 Shows the pH effect on the removal of acid dyes (initial concentration= 80 mg/L) within the pH range of 2-12 by the AC. At low pH values, the maximum percentage of dye removal is obtained, while at high pH values, the minimum percentage of dye removal is achieved. At lower pH values, more protons are available and activated carbon surface has positively charged site. As a result, there is electrostatic attraction between adsorbent and acid dyes. However, at higher pH values, the removal of acid dyes decreases due to the abundance of –OH groups in the solution and repulsion between activated carbon surface with a negative charge and acid dyes [14]. Similar results have been reported for the adsorption of acidic dyes on activated carbon [15-17]. The best of the adsorption efficiency for both dyes was obtained at pH 2, so all adsorbent experimental was performed under this pH by using hydrochloric acid solution.

3.2.2. Dye concentration effect

As shown in Figure 5, for both Acid Red 94 and Acid Blue 45, the amount of dye adsorbed onto AC increased with the initial dye concentration. Adsorption is a mass transfer process which is defined as the accumulation of material at the interface between two phases [18]. The effect of various initial dye concentrations of the two dyes was assessed (Figure 5). As can be seen, the higher the initial dye concentration, the lower is the percentage of dye adsorbed. The higher adsorption speed of Acid Red 94 in comparison with that of Acid Blue 45 can be attributed to the higher molecular weight of this dye and correspondingly, the stronger electrical and Van der Waals forces between the dye and the adsorbent.

3.3. Adsorption isotherm models

Establishing the most important correlation for equilibrium curve is an important factor to optimize the design of adsorption system to remove dyes from solution [2]. In the present study, four isotherm models were examined for their ability to describe the experimental data: the Langmuir, Freundlich, Temkin, and Dubinin–Radushkevich isotherms.

The most widely used model is Langmuir model which is given by [2]:

$$q_e = \frac{Q_0 K_L C_e}{1+K_L C_e} \quad (1)$$

where q_e, C_e, Q_0 and K_L are the amount of solute adsorbed at equilibrium (mg/g), the concentration of adsorbate at equilibrium (mg/L), maximum adsorption capacity (mg/g) and Langmuir constant (L/mg), respectively. Isotherm data were also tested using Freundlich isotherm which can be expressed as follows:

$$q_e = K_F\, C_e^{\frac{1}{n}} \quad (2)$$

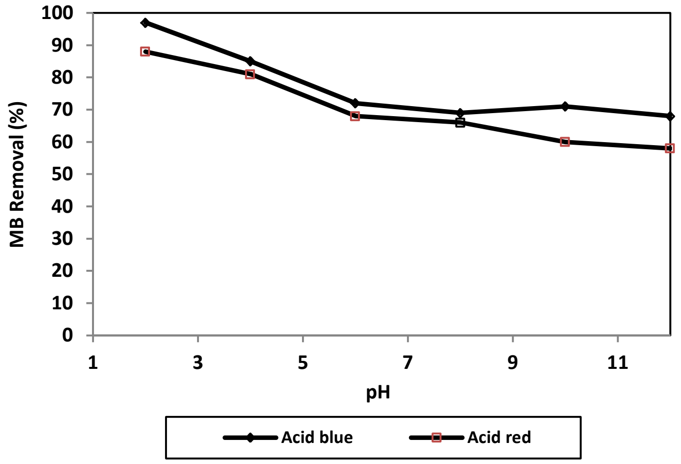


Figure 4: Effect of solution pH on Acid dyes removal by AC (dyes concentration= 80 mg/L, 0.5 g/L of AC, T=28 °C, S= 140 rpm).

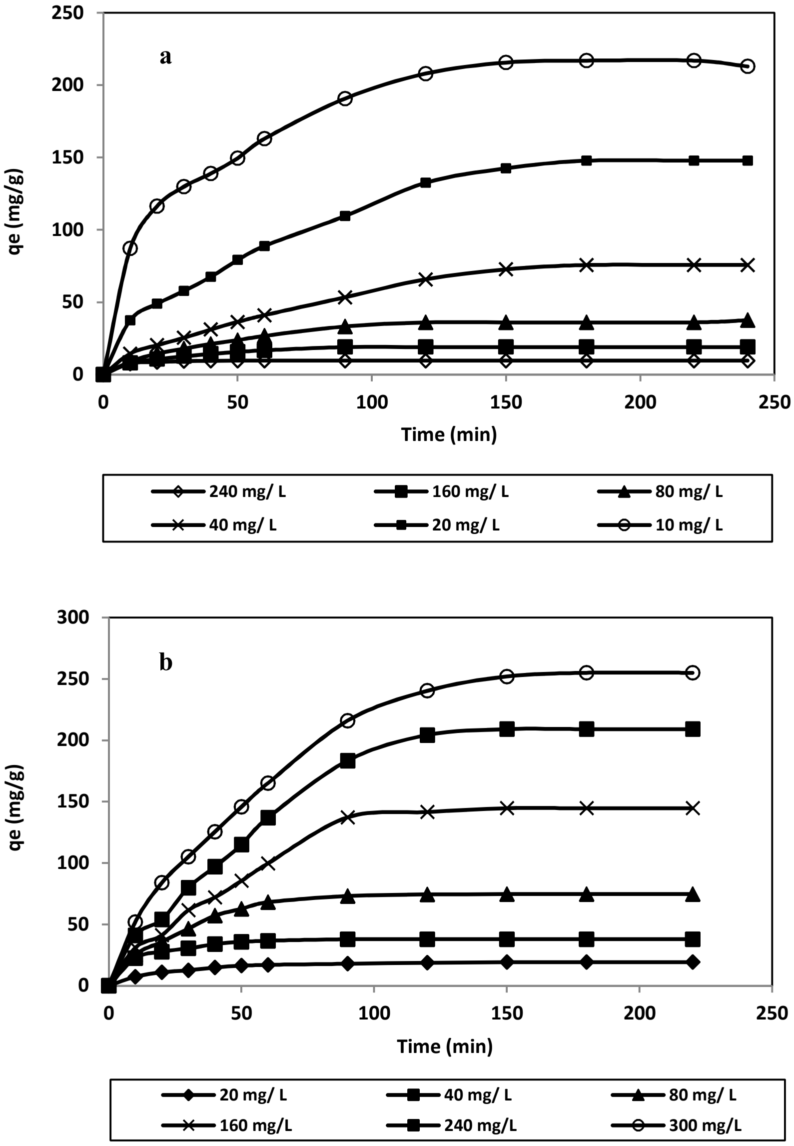


Figure 5: Effect of initial dye concentration (mg/L) on the adsorption of Acid Dyes by AC (WAC =0.5 g/L, T= 28 °C, S= 140 rpm, pH= 6) a: Acid Red 94 and b: Acid Blue 45.

Where K_F (mg/g) $(L/mg)^{1/n}$ and n are the Freundlich adsorption constants related to adsorption capacity and adsorption intensity, respectively.

The Tempkin isotherm is given as:

$$q_e = RT/b\ Ln\,(AC_e) \tag{3}$$

which can be linearized as:

$$q_e = BLn\,A + BLn\,C_e \tag{4}$$

Where

$$B_1 = RT / b \tag{5}$$

Where T and R are the absolute temperature (K) and the universal gas constant (8.314 J/mol K), respectively. The constant b is related to the heat of adsorption. Also, B_1 and K_T are the Tempkin constant and can be determined by a plot of q_e versus ln C_e [19].

The D–R equation is another popular isotherm model, which its derivation is not based on ideal assumptions, such as homogeneous surface or constant adsorption potential. It can give ideas about the physical or chemical nature of adsorption processes. The nonlinear form of D–R model is represented as Eq. (6) [4]:

$$q_e = q_m \exp(-\beta\varepsilon^2) \tag{6}$$

Where q_e and q_m are the quantity of adsorbed dye molecules per unit weight of adsorbent (mol/g) and the maximum adsorption capacity (mol/g), respectively. The Polanyi potential and can be calculated
By Eq. (7):

$$\varepsilon = RT\ln(1+\frac{1}{C_e}) \quad (7)$$

where R is the gas constant (8.314 J/mol K) and T is the absolute temperature. The constant β (mol^2/J^2) is the activity coefficient corresponding to adsorption mean free energy, E (kJ/mol), which can be determined by the following relationship:

$$E = \frac{1}{(2\beta)^{0.5}} \quad (8)$$

The conformity of isotherm models to experimental data was evaluated by correlation coefficient (R^2) and normalized standard deviation, (Δq_e) %, defined by Eq. (9) [4]

$$\Delta q_e(\%) = \frac{\sqrt{\Sigma[\frac{q_{e,exp}-q_{e,cal}}{q_{e,exp}}]^2}}{N-1} \times 100 \quad (9)$$

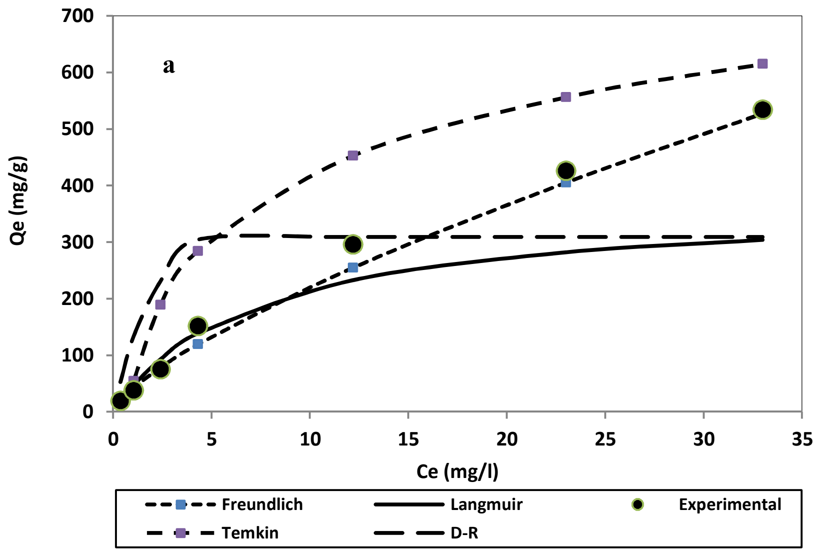
Where $q_{e,exp}$ (mg/g) and $q_{e,cal}$ (mg/g) are the experimental and calculated values for the amount of dyes adsorbed, respectively. Small values of Δq_e represent that the data from the model is similar to the experimental values, whereas larger Δq_e values point out more difference between the model and experimental data [4].

A plot of q_e against C_e as a fitted curve is shown in Figure 6. The Langmuir, Freundlich, Tempkin and Dubinin–Radushkevich (D–R) constants were derived from the adsorption data (Table 1). In comparison with other models, The Freundlich model for both acidic dyes with the highest values of correlation coefficients and the lowest values of ΔQ_e describe the adsorption process well.

The evaluation of the adsorption capacity of the acid dyes by K_F parameter of the Freundlich model and the q_m parameter of Langmuir model shows that the adsorption capacity of Acid Blue 45 is higher than Acid Red 94. In this case, the smaller molecular weight and the size of Acid Blue dye, compared to those of Acid Red 94, could allow more dye molecules to be deposited in the microporous pores of the activated carbon, resulting in greater absorption capacity. Many research works in the literature have also reported that Ferdinand model is an appropriate model describing the absorption process for activated carbon derivatives [20].

Table 1: Langmuir, Freundlich, Temkin and Dubinin-Radushkevich (D-R) isotherm model parameters for the adsorption of Acid Dyes (Acid Red 94 and Acid Blue 45) onto WCAC.

Isotherm	Dye	q_m (mg/g)	b (L/mg)	R^2	Δq_e
Langmuir	Acid Red 94	370.475	0.138	0.97950	27.94
	Acid Blue 45	434.78	0.130	0.98220	22.79
Frendlich	-	K_F (mg/g)	1 / n	R^2	Δq_e
	Acid Red 94	40.945	0.73	0.99010	9.91
	Acid Blue 45	49.650	0.60	0.99410	9.17
Tempkin	-	k_T	B	R^2	Δq_e
	Acid Red 94	1.32	15.32	0.94840	46.99
	Acid Blue 45	2.30	76.77	0.98340	36.00
D-R	-	q_m (mg/g)	E (KJ/mol)	R^2	Δq_e
	Acid Red 94	342.96	0.158	0.9267	10.73
	Acid Blue 45	324.54	0.400	0.9876	69.64



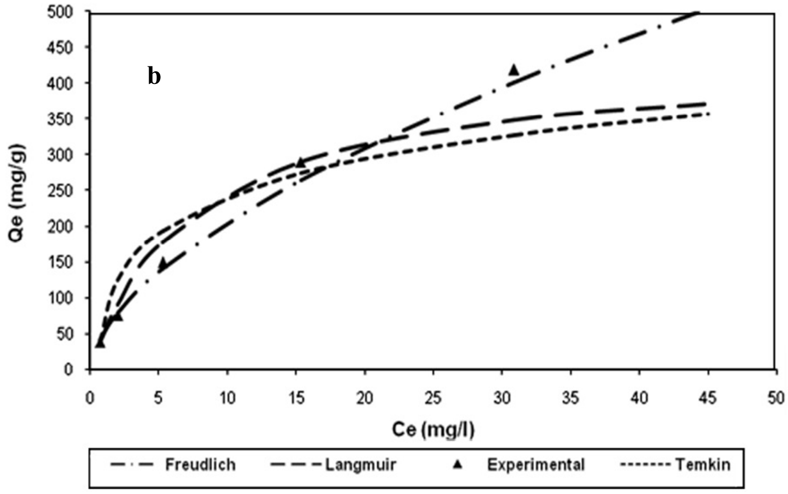


Figure 6: The non-linear isotherms studied for adsorption of Acid Dyes on AC (WAC =0.5 g/L, T= 28 °C, S= 140 rpm, pH= 6) a: Acid Red 94 and b: Acid Blue 45.

3.4. Adsorption kinetics models

Several kinetic models can be used to express the mechanism of solute sorption onto a sorbent. In order to design a fast and effective model, investigations were made on adsorption rate. For the examination of the controlling mechanisms of the adsorption process, such as chemical reaction, diffusion control and mass transfer, several kinetic models are used to assess the experimental data [2].

The pseudo-first-order model proposed by Lagergren and Svenska [21] is presented by Eq. (10):

$$log(q_e - q_t) = log\, q_e - k_1 t/2.303 \tag{10}$$

Where q_e and q_t are the amount of dye adsorbed (mg/g) at equilibrium and time t (min), respectively. k_1 is the rate constant of adsorption (min^{-1}).

Data were applied to the pseudo-second order kinetic rate equation which is expressed as [22]:

$$^{t}/_{q_t} = {}^{1}/_{k_2 q_e^2} + {}^{t}/_{q_e} \tag{11}$$

Where k_2 is the equilibrium rate constant of pseudo-second order (g/mg min). The possibility of intraparticle diffusion resistance affecting adsorption was explored by using the intraparticle diffusion model as

$$q_t = k_p t^{\frac{1}{2}} + I \tag{12}$$

Where k_p (mg/g $min^{1/2}$), the intraparticle diffusion rate constant, is obtained from the slope of the straight line of q_t vs. $t_0^{.5}$ and C is the intercept.

The kinetics of acid dyes adsorption on activated carbon was studied by the pseudo-first-order model and pseudo-second-order model. The calculated kinetic data and models with the empirical data are presented in Figure 7 and the parameters of the models are shown in Tables 2.

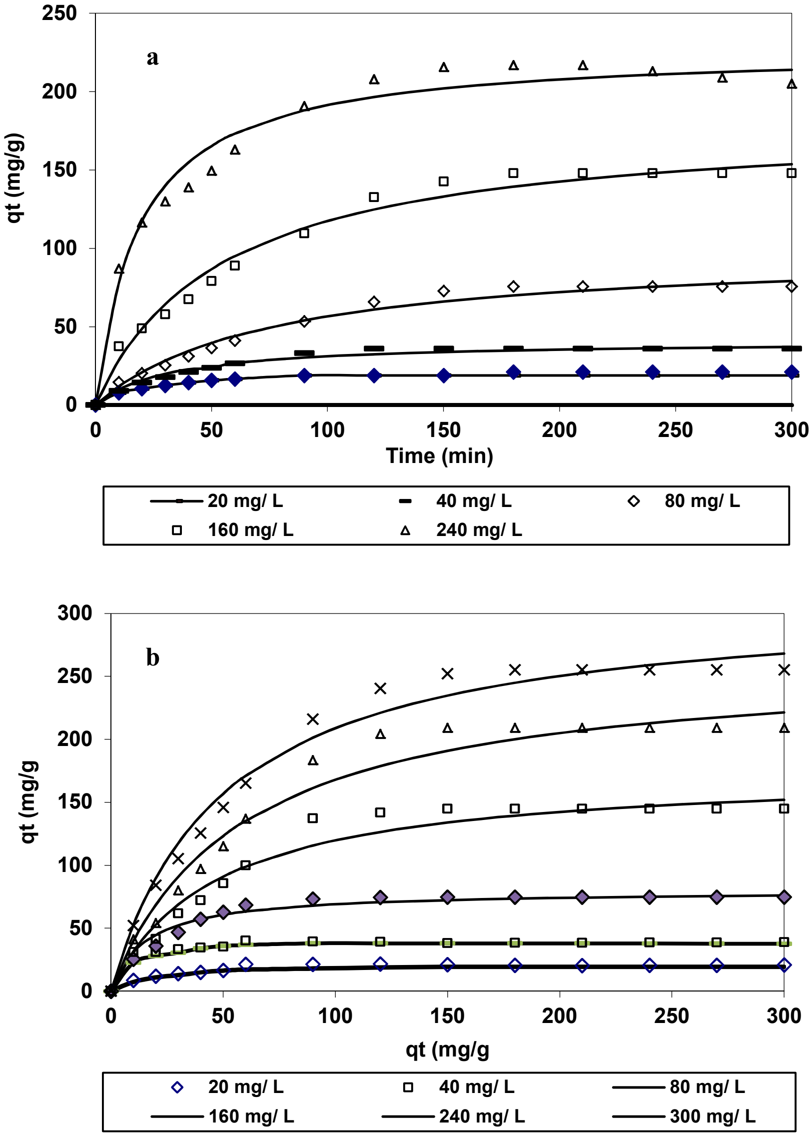


Figure 7: Pseudo-second order sorption kinetics of dye on AC (WAC =0.5 g/L, T= 28 °C, S= 140 rpm, pH= 6) a: Acid Red 94 and b: Acid Blue 45.

Table 2: Kinetic parameters of pseudo-first and pseudo-second-order models for the adsorption of Acid Red 94 and Acid Blue 45 on WCAC.

Initial dye concentration	$q_{e,,exp}$ (mg/L)		Pseudo- first order			Pseudo- first order			
		$q_{e,,cal}$ (mg/$_L$)	k_1 (min^{-1})	R^2	Δq_t	$q_{e,,cal}$ (mg/L)	$k_2 * 10^3$ (min^{-1})	R^2	Δq_t
Acid Red 94									
10	9.62	9.24	0.19	0.992	10.82	9.75	85.34	0.999	6.32
20	18.94	17.98	0.36	0.995	7.95	21.18	37.08	0.998	7.22
40	36.01	32.64	1.03	0.996	27.92	37.19	0.76	0.993	6.92
80	75.68	85.34	2.12	0.996	13.85	79.23	0.13	0.982	8.50
160	14.80	153.22	2.92	0.971	22.61	153.72	0.10	0.988	9.46
240	20.99	201.89	3.92	0.947	21.17	213.90	0.23	0.994	6.78
Acid Blue 45									
20	18.94	18.09	0.64	0.994	14.58	20.65	3.436	0.999	11.69
40	36.01	34.23	1.93	0.980	14.04	38.80	5.302	0.999	6.55
80	75.68	75.61	2.71	0.968	14.19	75.96	0.784	0.998	10.25
160	147.80	146.96	2.54	0.940	8.35	151.88	0.122	0.984	11.04
240	204.99	219.00	3.98	0.924	11.03	221.25	0.066	0.980	10.98
300	255.41	262.87	5.19	0.972	7.79	268.07	0.063	0.990	6.75

Correlation coefficient values (R^2) at different concentrations of both acid dyes for both models are greater than 0.90. However, the pseudo-second-order model with relatively large correlation coefficients (close to unit) and smaller Δq_e values in the whole concentration range of the studied cases showed better fit with experimental data. Accordingly, the pseudo-second-order kinetics model describes the process of adsorption of acid dyes and shows that the overall rate of adsorption process is controlled by chemical absorption.

The intraparticle diffusion kinetics models from process of adsorption acid dyes on activated carbon was also studied (Table 3). If penetration in the cavities is the velocity controlling stage of the adsorption process, the drawing q versus $t^{0.5}$ will be in the form of a straight line with a slope K_{ip} and the width of the source zero.

At the first part of the plot, according to y-intercept is not equal to zero (Figure 8), therefore, the total adsorption rate in this step is not controlled by intraparticle penetration and controlled by penetration in the film layer or by chemical adsorption. In the next step, penetration into the pores controlling the adsorption velocity is adsorbed.

By increasing the concentration of both dyes in the solution phase, the traction forces of the dye molecules to the adsorbent pores and subsequently the parameters of the penetration rate at the film layer as well as the absorbent pores increase. The higher the Acid Red speed than Acid Blue can be attributed to the smaller molecular dimensions and lower inhibition of Acid Red penetration.

Table 3: Diffusion coefficients for adsorption of Acid Dyes on WCAC.

Acid Blue				Acid Red		
Initial dye concentration (mg/L)	$k_{i,p}$ (mg/g min$^{0.5}$)	C	R^2	$k_{i,p}$(mg/g min$^{0.5}$)	C	R^2
20	1.49	3.14	0.928	1.73	4.25	0.942
40	3.55	16.46	0.920	2.52	1.56	0.993
80	6.66	1.28	0.943	8.29	9.55	0.989
160	12.43	30.15	0.978	17.11	7.20	0.994
240	15.85	36.67	0.992	21.94	39.14	0.998
300	20.87	27.65	0.998	24.57	52.59	0.992

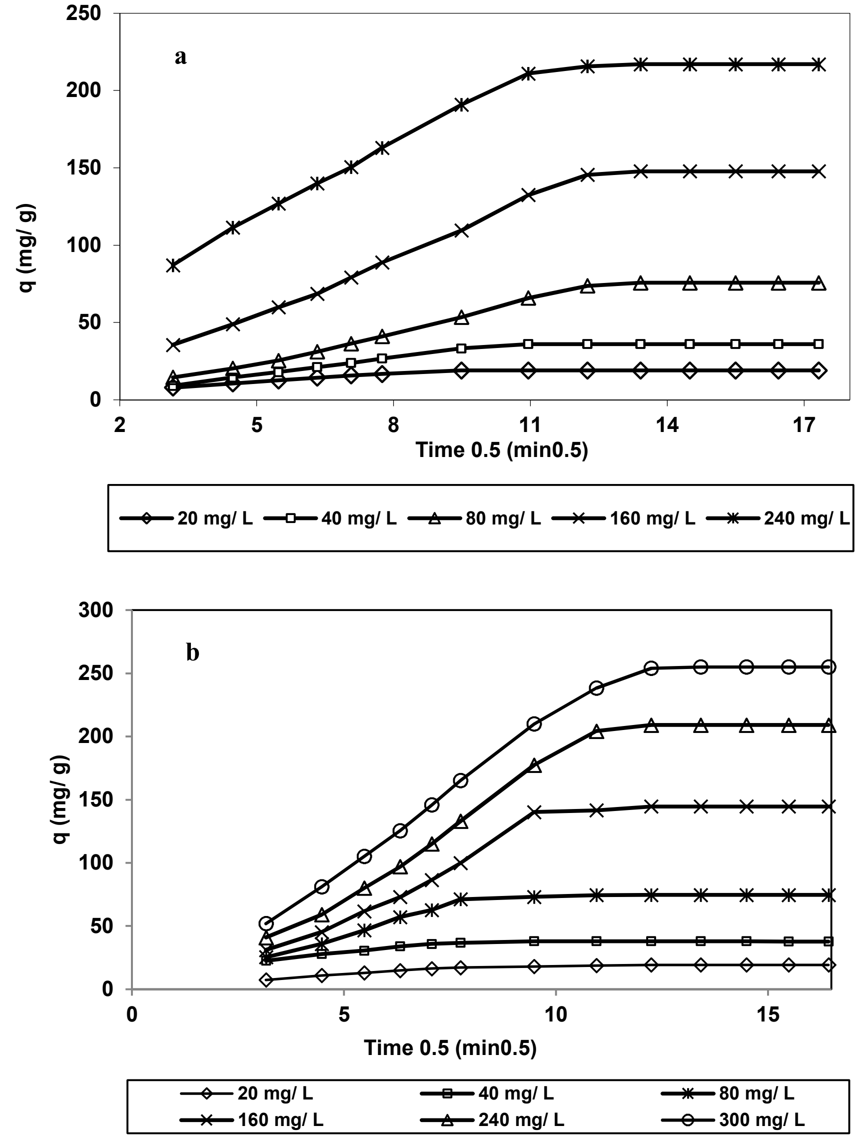


Figure 8: Intraparticle diffusion plot for the adsorption of different initial Acid Dyes concentrations (WAC = 0.5 g/L, T= 28 °C, S= 140 rpm, pH= 6)a: Acid Red 94, b: Acid Blue 45.

Table 4: Thermodynamic parameters at various temperatures.

Dye	Standard enthalpy ΔH^o (kJ/mol)	Standard entropy ΔS^o (J/mol K)	Gibbs free energy ΔG^o (kJ/mol)		
			303 K	313 K	323 K
Acid Red	-3.87	36.24	-5.724	-6.233	-6.269
Acid Blue	-2.54	41.71	-3.423	-3.564	-4.012

3.5. Thermodynamic parameters

Thermodynamic parameters including the changes in Gibbs free energy (ΔG), enthalpy (ΔH), and entropy (ΔS) are the actual indicators for practical application of an adsorption process. According to the values of these parameters, it can be determined what process will occur spontaneously. ΔG can be calculated using Eq. (13). ΔH and ΔS were calculated from the slope and intercept of the linear plot of ln (qe/Ce) vs. 1/T [23].

The equilibrium experiments were conducted for 200 mg/L Acid Dyes solution at 30, 40, and 50°C and the obtained thermodynamic parameters. The results of these experiments are presented in Table 4. The negative values obtained for changing the Gibbs free energy shows that the adsorption process is favorable and spontaneous. In addition, increasing the temperature results in increasing the degree of spontaneity of the reaction. According to Table 4, it is indicated that the ΔS values are positive which can be occurred as a result of increased randomness or excitement at the interface (solid–liquid) during the adsorption of Acid dyes on WCAC. Furthermore, the positive value of ΔH° signifies that the adsorption process is exothermic in nature [4].

$$\Delta G=-RT\ln\left(q_e/C_e\right) \qquad (13)$$

4. Conclusions

In the present study, applicability of activated carbon (AC) to remove Acid Dyes from aqueous solutions was investigated. The experimental results indicated that AC is effective for the removal of Acid Dyes from aqueous solution. The isotherm data of dyes followed Freundlich isotherm. Adsorption kinetic of dyes was studied for both Acid Dyes and rate sorption was found to conform to pseudo-second order kinetic model. Investigation of thermodynamic parameters indicated that the adsorption process is spontaneous and exothermic in nature. The experimental results revealed that the produced activated carbon has developed porous structure, fibrous shape, predominantly amorphous structure and could be employed as a low-cost and effective adsorbent in the removal of Acid Dyes from wastewater.

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