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Modification of activated carbon by the alkaline treatment to remove the dyes from wastewater: mechanism, isotherm and kinetic

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ABSTRACT

The present paper investigates the surface modification of activated carbon (AC) by alkaline (NaOH) and dye removal ability of the surface modified activated carbon (SMAC). Acid Red 14 (AR14) and Acid Blue 92 (AB92) were used as the dye models. The surface characteristics of SMAC were investigated using the Fourier transform infrared (FTIR), scanning electron microscopy (SEM), and surface area. The possible mechanism of the adsorption process and dye interaction with SMAC surface was analyzed. The effects of adsorbent dosage, initial dye concentration, and pH on the dye removal were investigated. The isotherm and kinetic of dye adsorption were studied. The adsorption isotherm of the dyes onto AC and SMAC followed Langmuir and Freundlich isotherms, respectively. The kinetic of dyes onto both AC and SMAC followed pseudo-second-order kinetic model. The results indicated that the alkaline (NaOH) treatment of AC was an efficient method to modify the AC. The maximum adsorption capacity (Q_{max}) of AR14 and AB92 was 2.50 and 0.69 mg/g onto AC and 9.17 and 11.77 mg/g onto SMAC, respectively. Dye desorption tests (SMAC regeneration) showed that the maximum dye release of 85% for AR14 and 83% for AB92 was achieved in an aqueous solution at pH 12. In addition, the SMAC could be used as an eco-friendly adsorbent to remove the dyes from colored wastewater.

Keywords: Surface modification; Activated carbon; Alkaline treatment; Dye removal; Isotherm and kinetic

1. Introduction

Synthetic dyes are an important class of organics and are often found in the environment as a result of their wide spread industrial use. These compounds are difficult to degrade owing to their complex aromatic structure and synthetic origin. Many of them are known to be toxic or carcinogenic. Therefore, there is a definite need to have a technology, which may work suitably under the above circumstances and should be cost effective for the treatment of colored wastewater [1–6]. Several methods, such as adsorption [7–9], nanofiltration [10], ozonation [11], and electroflotation [12] were used to remove the organics from the wastewater. Among these processes, adsorption was found to be superior to other techniques employed for wastewater treatment in terms of initial cost, simplicity of design, ease of operation, and insensitivity of toxic substances [13].

Activated carbon (AC) is the most commonly used and most effective adsorbent [14]. Nevertheless, its application is restricted due to its high cost. The use of low-cost agriculture by-products to produce AC has been shown to provide an economical solution to this problem [15,16]. The adsorption of pollutants by

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AC is being widely used to treat wastewater and the advantages of this adsorbent have been well documented [17,18].

A large number of publications deal with the removal of pollutants in aqueous solutions by the AC. In addition, the performance of the AC is occasionally interpreted only in terms of their physical properties (surface area, porosity, etc.), not taking into account the influence of its surface chemistry, which may be the key to performance optimization. The nature of the surface functional groups can be modified through physical, chemical, and electrochemical treatments at different temperatures, pressures, concentrations, and contact times [19,20].

In the present paper, the surface modification of AC by alkaline (NaOH) treatment and dye removal ability of surface modified activated carbon (SMAC) was investigated. Acid Red 14 (AR14) and Acid Blue 92 (AB92) were used as the dye models. The surface characteristics of SMAC were investigated using the Fourier transform infrared (FTIR), scanning electron microscopy (SEM), and surface area (Brunauer–Emmet–Teller: BET method). The effects of adsorbent dosage, initial dye concentration, and pH on dye removal were investigated. The isotherm and kinetic of dye adsorption onto AC and SMAC were studied. In addition dye desorption studies (adsorbent regeneration) carried out.

2. Experimental

2.1. Chemicals

Acid dyes (AR14 and AB92) were obtained from Ciba Ltd. and used without further purification. The chemical structure of the dyes is shown in Fig. 1. All other chemicals were of analytical grade and were purchased from Merck (Germany).

2.2. Surface modification of AC

NaOH (0.5 mol) was weighed and placed into a bottle containing 1 L distilled water. The bottle was mixed constantly for 10 min. and 10 g of AC was added to the bottle. The bottle was mixed once again constantly for 1 h. The modified AC was then separated, washed, and placed in an oven at 100˚C and dried for 4 h then transferred to a desiccator until use.

2.3. Surface characterization

The surface functional groups of AC and SMAC were studied by the FTIR spectroscopy. The FTIR spectra were recorded between 400 and 4,000 cm^{-1} using the Perkin-Elmer Spectrophotometer Spectrum 1 Net.

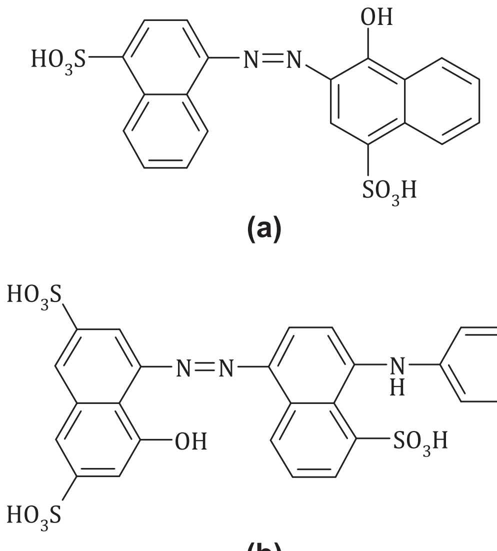

Fig. 1. The chemical structure of dyes (a) AR14 and (b) AB92.

To know the structure of AC and SMAC, SEM was generally employed to visualize sample morphology using a Philips XL30 microscope.

Specific surface area of the AC and SMAC was evaluated through N_2 adsorption at 77 K, using an Autosorb-iQ-Quantachrome instrument. The BET (Brunauer–Emmet–Teller) model was applied.

2.4. Adsorption procedure

The dye adsorption measurements were conducted by mixing various amounts of AC and SMAC (0.3–1.2 g) in jars containing 250 mL of a dye solution (50 mg/L). The solution pH was adjusted by adding a small amount of H_2SO_4 or NaOH. The dye solutions were prepared using distilled water to prevent and minimize possible interferences in this study.

The effect of pH on the dye removal was investigated by contacting 250 mL of dye solution with an initial dye concentration of 50 mg/L using the jar test at room temperature (25˚C) and 1.2 g of adsorbent.

The effect of adsorbent dosage (AC/SMAC) on dye removal was investigated by contacting 250 mL of dye solution with an initial dye concentration of 50 mg/L using the jar test at room temperature (25˚C) and pH 2 for 30 min. Different amounts of the adsorbent (0.3–1.2 g) for AR14 and AB92 were applied. After equilibrium, the samples were centrifuged and the concentration in the supernatant dye solution was analyzed.

The effect of initial dye concentration on the percentage of dye removal was studied. The adsorbent (1.2 g) was added to 250 mL at different dye concentrations of 25, 50, 75, and 100 mg/L. These experiments were performed at pH 2.

The maximum wavelength (λ_{max}) of dyes to determine the residual dye concentration in solution was 517 nm for AR14 and 595 nm at acidic and neutral pH values and 571 nm at basic pH values for AB92. The changes in absorbance were determined at certain time intervals (2.5, 5, 7.5, 10, 15, 20, 30, 40, 50, and 60 min) during the adsorption process. After the experiments, the samples were centrifuged by Hettich EBA20 and then the dye concentration was determined. UV–vis spectrophotometer CECIL 2021 was employed for conducting absorbance measurements of samples. The results were verified with the adsorption isotherms (Langmuir, Freundlich and Temkin) and kinetics (pseudo-first-order, pseudo-second-order and intraparticle diffusion models).

2.5. *Dye desorption and SMAC regeneration studies*

The SMAC used for the adsorption of 50 mg/L of dye solution was separated from the solution by centrifugation and dried. Then the adsorbent was agitated with 250 mL of distilled water at pH 12 for the predetermined equilibrium time of the desorption process. The desorbed dye was determined.

3. Results and discussion

3.1. *Surface characteristics of AC and SMAC*

The FTIR spectra show that the peak positions are at 3,409, 2,923, 2,845, 1,600, 1,171, 1,094, and 1,042 cm^{-1} for AC and 3,430, 2,916, 2,855, 1,611, 1,171, and 1,034 cm^{-1} for SMAC (Fig. 2). The band at 3,409 cm^{-1} for AC (3,430 cm^{-1} for SMAC) is due to O–H and N–H stretchings. The bands at 2,923 and 2,845 cm^{-1} for AC (2,916 and 2,855 cm^{-1} for SMAC) correspond to unsymmetrical and symmetrical aliphatic C–H stretchings, respectively. While the band at 1,600 cm^{-1} for AC (1,611 cm^{-1} for SMAC) reflects the C=C stretching of aromatic rings whose intensity is enhanced by the presence of oxygen atoms as phenol or ether groups, bands at 1,300–1,000 cm^{-1} correspond to C–O stretching [21,22].

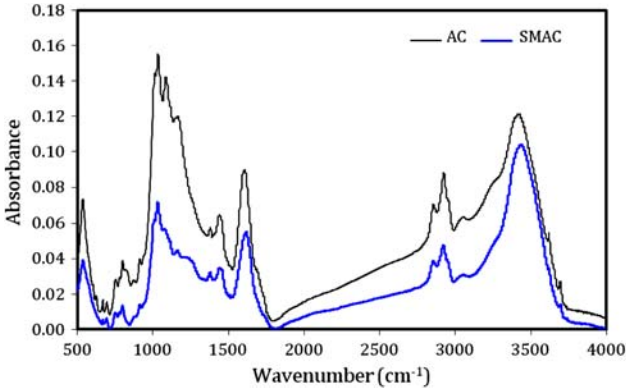

Fig. 2. FTIR spectrum of AC and SMAC.

The surface of AC is thought to be assemblies of hexagon structure and the reaction site is at the carbon–oxygen bond. FTIR studies showed that the peaks' positions of AC and SMAC did not change significantly but the intensity of peaks changed clearly after the surface modification of AC. The intensity of peaks at 3,409–3,430 cm^{-1} increased after surface modification due to the increase of the hydroxyl groups. It can be attributed to the formation of hydroxyl groups towing to the reaction of carbon–oxygen bond and alkaline. The results show that the alkaline treatment is an effective method to modify the AC surface.

The scanning electron micrographs of AC and SMAC are shown in Fig. 3. The SEM image of AC appears to have a smoother surface than the SMAC.

The surface area (S_{BET}) of the AC and SMAC was 116 and 96 m^2/g, respectively. While the alkaline treatment of AC displays beneficial effects on the adsorption of pollutants, it is, however, detrimental to the physical aspects of AC such as the BET surface area. The alkaline treatment reduces the BET surface area of AC. It may be attributed to the destruction of the porous structure within the AC [23]. Under alkaline treatment, it is expected that the hydroxide anion will react with the surface functional groups of AC. This was verified in the previous published paper where AC treated with NaOH showed a major increase in the concentration of phenolic functional groups on the surface [19,23].

3.2. *pH effect on the dye removal and dye adsorption mechanism*

The effect of initial pH on dye removal is shown in Fig. 4. The dye adsorption capacity of AC does not change significantly when the pH changes. It can be attributed that hydrophobic groups' interactions are the dominant mechanism for dye adsorption by the AC. But the dye adsorption capacity of SMAC increases considerably when the pH decreases. SMAC is comprised various oxygen functional groups such as

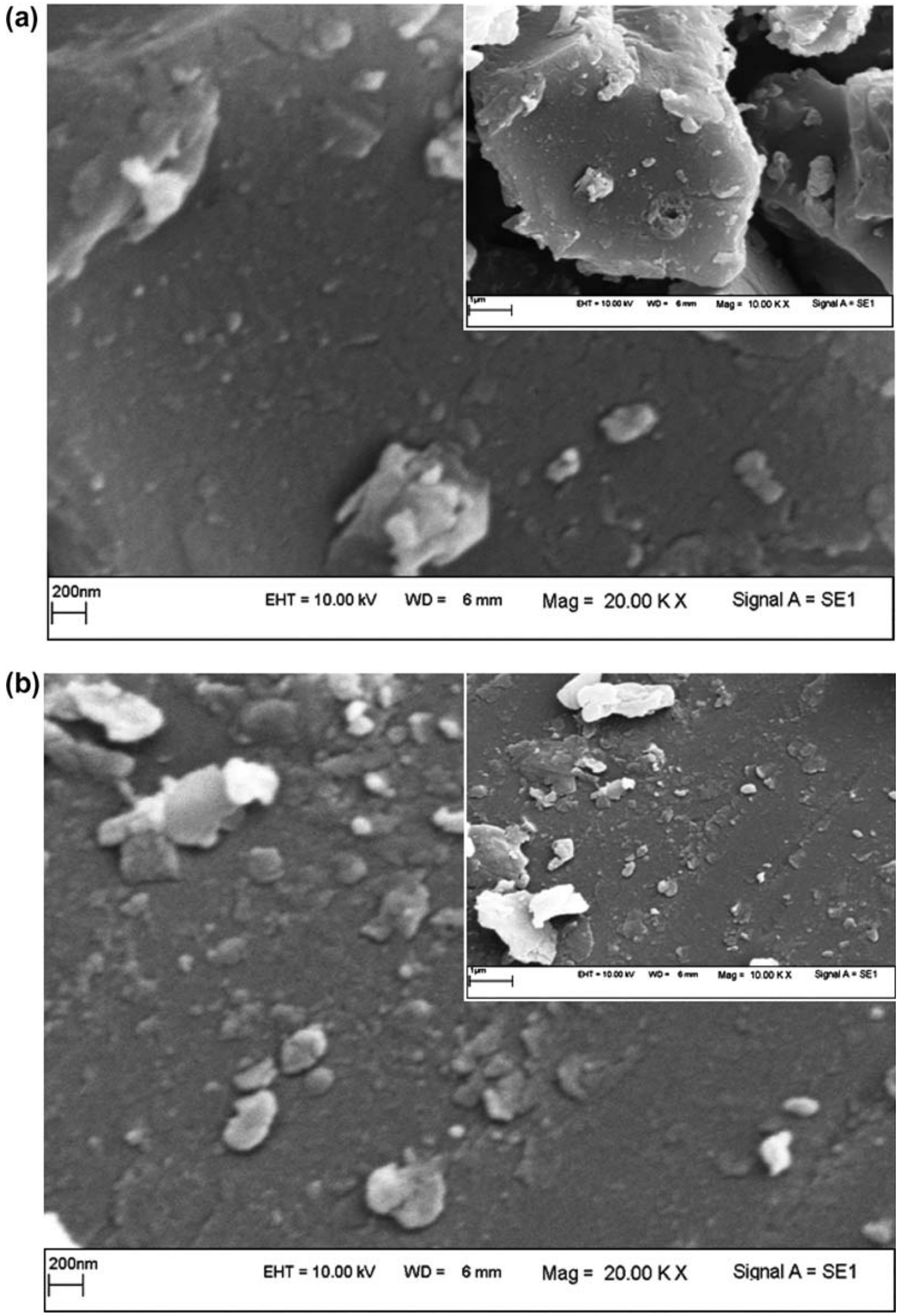

Fig. 3. SEM images (a) AC and (b) SMAC.

hydroxyl and carboxyl groups than that of AC which are affected by the solution pH. Thus, the electrostatic attraction is the dominant mechanism for dye adsorption by SMAC. Therefore, at various pH values, the ionic properties and structure of dye molecules could play a very important role in the dye adsorption on

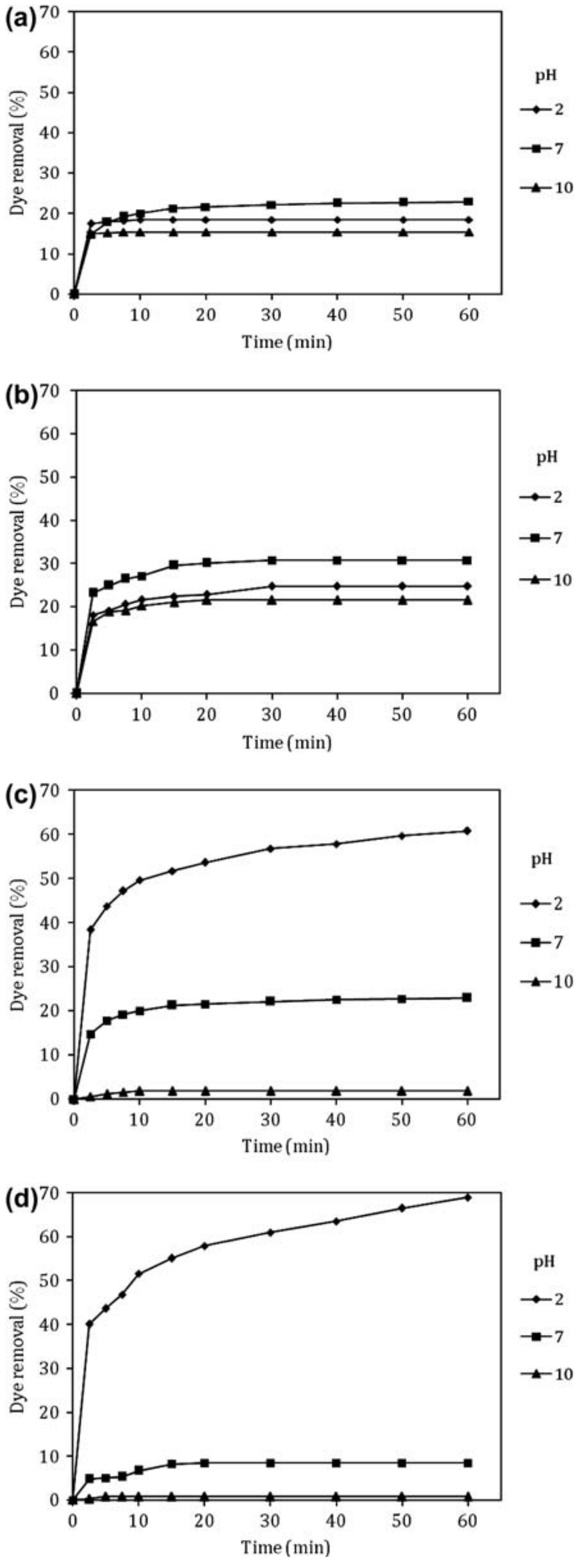

Fig. 4. Effect of pH on dye removal by AC (a: AR14 and b: AB92) and SMAC (c: AR14 and d: AB92) at different time intervals.

SMAC. At pH 2, a considerably high electrostatic attraction exists between the positively charged surfaces of the adsorbent, due to the ionization of functional groups of the SMAC and negatively charged anionic dye molecules. The pK_a values for AR14 and AB92 are 6.7 and 7, respectively. The proposed mechanism of dye adsorption by SMAC at pH 2 is shown in Fig. 5. As the pH of the system increases, the number of negatively charged sites on SMAC increases. A negatively charged site on the adsorbent does not favor the adsorption of anionic dyes due to electrostatic repulsion. Thus, the effective pH for SMAC was 2, and it was used in further studies.

3.3. Adsorbent dosage effect on dye removal

Fig. 6 shows the influence of adsorbent dosage on the dye removal at pH 2. The percentage removal increased with the adsorbent dosage. The increase in the adsorption of dyes with adsorbent dosage was due to the availability of more active surface sites of the adsorbent for adsorption. The dye adsorption capacity of SMAC increases considerably than that of AC when the adsorbent dosage increases. SMAC is comprised more various oxygen functional groups which are affected by the pH of solution. When the adsorbent dosage increases, a considerably higher electrostatic attraction exists between the positively charged surfaces of the adsorbent at pH 2.The optimum adsorbent dosage for AR14 and AB92 was 1.2 g of AC/SMAC for 250 mL of 50 mg/L dye solution.

3.4. Dye concentration effect on dye removal

Fig. 7 shows the effect of dye concentration on dye removal by AC and SMAC (pH 2). The amount of dye adsorbed onto the adsorbent increased with an increase in the initial dye concentration of solution if the amount of adsorbent was kept unchanged. It can be attributed to an increase in the driving force of the concentration gradient with a higher initial dye concentration. The dye adsorption is very intense and reaches equilibrium very quickly at low initial concentration. It shows the possibility to form a monolayer coverage of the adsorbate molecules at the adsorbents outer interface of the adsorbent. The adsorbed amount of dye increased with increasing concentration of the solution at a fixed adsorbent dosage, but the percentage of adsorption decreased. In other words, the residual concentration of dye molecules will be higher for higher initial dye concentrations. The ratio of the initial number of dye molecules to the available adsorption sites is low and

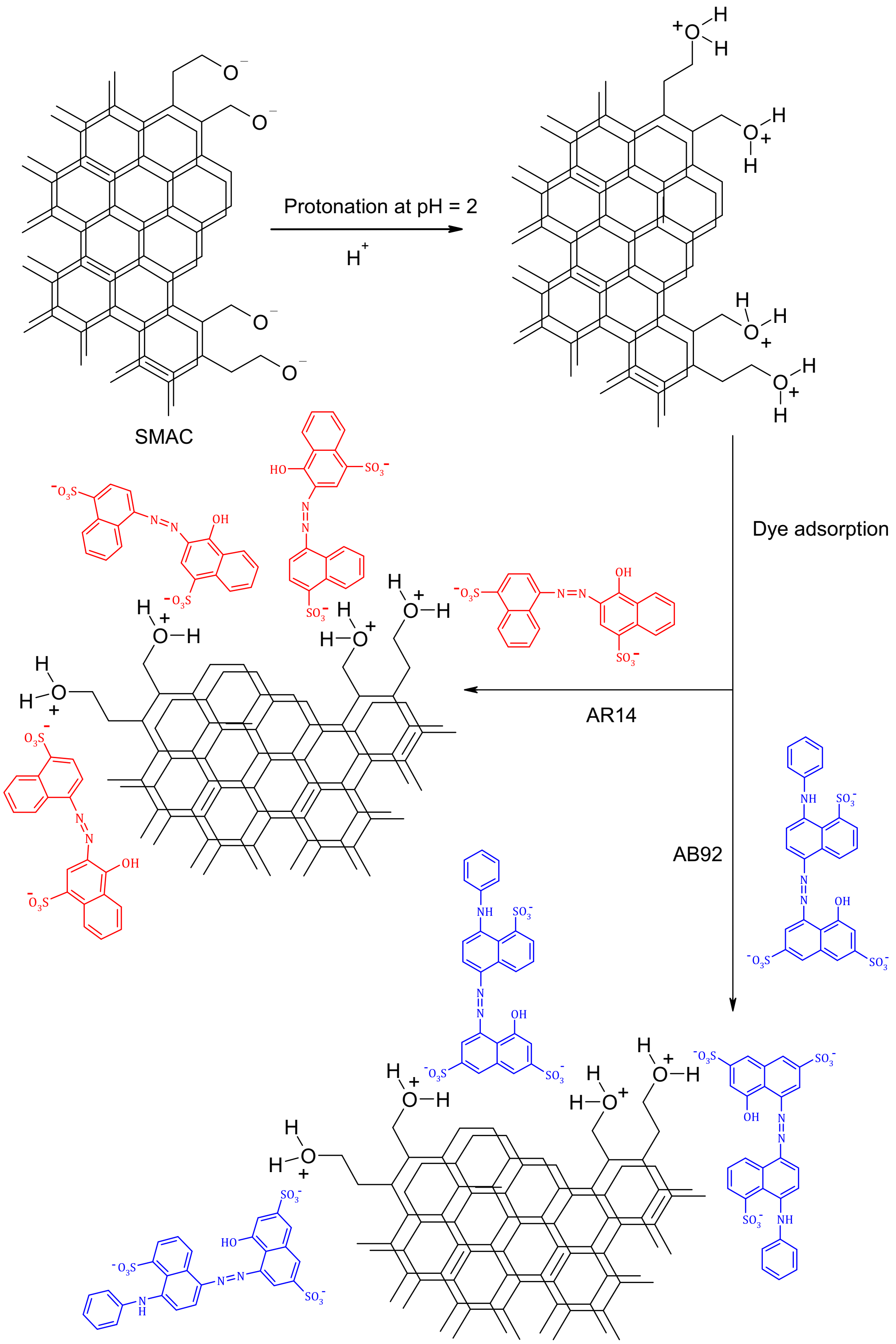

Fig. 5. The proposed mechanism of dye adsorption by SMAC.

subsequently the fractional adsorption becomes independent of the initial concentration at lower adsorbate concentrations. At higher concentrations, however, the number of available adsorption sites becomes lower and subsequently the removal of dyes depends on the initial concentration [24,25].

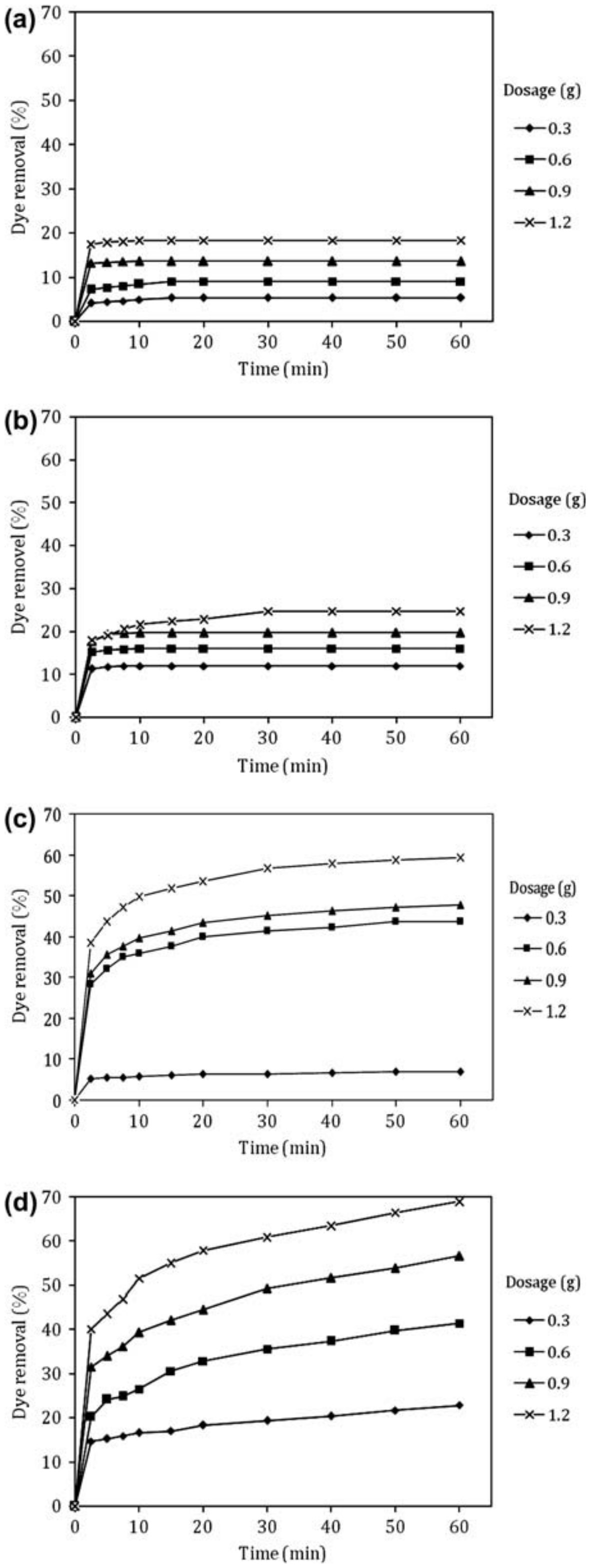

Fig. 6. Effect of adsorbent dosage on dye removal by AC (a: AR14 and b: AB92) and SMAC (c: AR14 and d: AB92) at different time intervals.

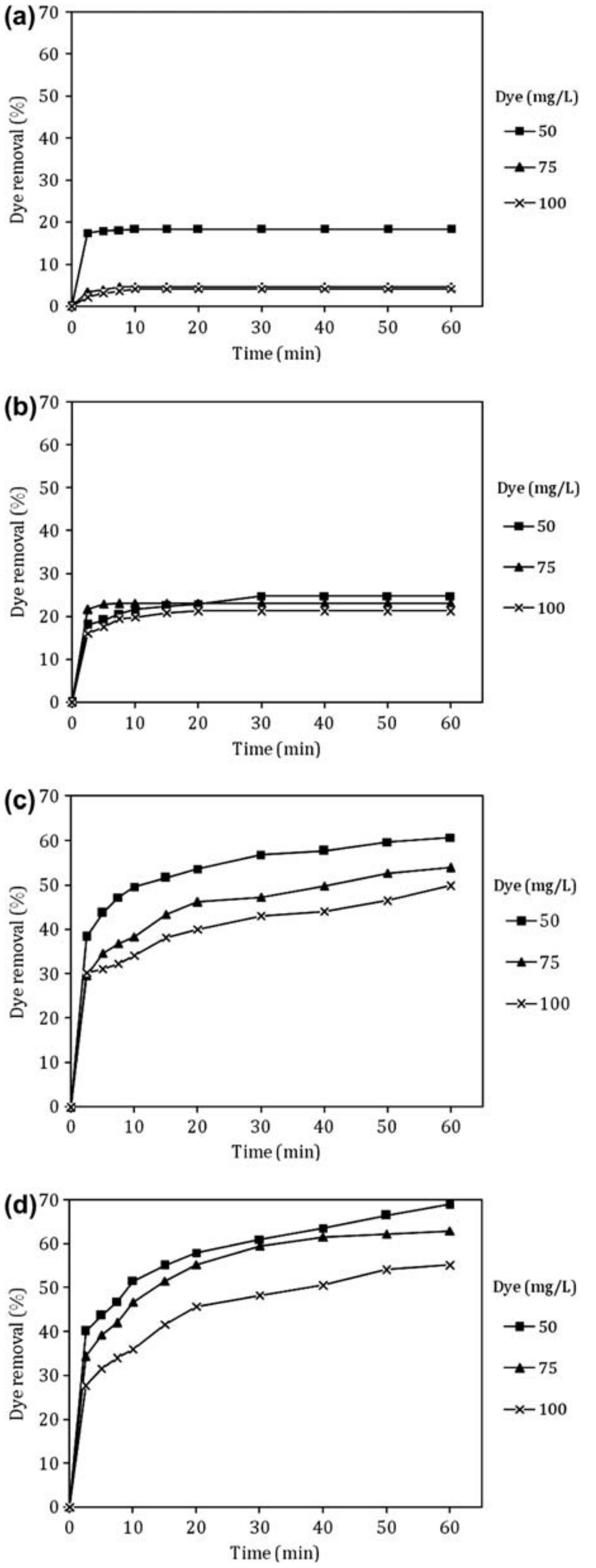

Fig. 7. Effect of initial dye concentration (mg/L) on dye removal by AC (a: AR14 and b: AB92) and SMAC (c: AR14 and d: AB92) at different time intervals.

3.5. Adsorption isotherm

It is important to establish the most appropriate correlation for the equilibrium curve to optimize the design of an adsorption system to remove pollutants from solutions. Several models have been used in the literature to describe the experimental data of adsorption isotherms. The Langmuir model is the most frequently employed model and is given by [26]

$$\frac{C_e}{q_e} = \frac{1}{K_L Q_{max}} + \frac{C_e}{Q_{max}} \qquad (1)$$

where q_e, C_e, Q_{max}, and K_L are the amount of dye adsorbed at equilibrium (mg/g), concentration of adsorbate at equilibrium (mg/L), maximum adsorption capacity (mg/g), and Langmuir constant (L/mg), respectively.

Essential characteristics of the Langmuir isotherm can be expressed by a dimensionless constant called equilibrium parameter, R_L, which is defined by the following equation [27]:

$$R_L = \frac{1}{1 + K_L C_0} \qquad (2)$$

where C_0 is the initial dye concentration (mg/L). The nature of the adsorption process is to be either unfavorable ($R_L > 1$), linear ($R_L = 1$), favorable ($0 < R_L < 1$), or irreversible ($R_L = 0$). Values of R_L indicated that the adsorption processes were favorable.

To study the applicability of the Langmuir isotherm for the dye adsorption onto AC and SMAC, the linear plot of C_e/q_e against C_e is plotted. The values of Q_{max}, K_L, R_L and R^2, (correlation coefficient values of all isotherm models) are shown in Table 1.

The Freundlich isotherm is derived by assuming a heterogeneous surface with a non-uniform distribution of heat of adsorption over the surface. Freundlich isotherm can be expressed by [28–31]:

$$q_e = K_F C_e^{1/n} \qquad (3)$$

where K_F is the adsorption capacity at unit concentration and $1/n$ is the adsorption intensity. $1/n$ values indicate the type of isotherm to be irreversible ($1/n = 0$), favorable ($0 < 1/n < 1$), unfavorable ($1/n > 1$) [28]. Eq. (3) can be rearranged to a linear form:

$$\log q_e = \log K_F + (1/n) \log C_e \qquad (4)$$

To study the applicability of the Freundlich isotherm for the dye adsorption onto AC and SMAC, linear plots of log q_e vs. log C_e are plotted. The values

Table 1
Isotherm constants for dye adsorption onto AC and SMAC at different adsorbent dosages (200 mL solution, T 25°C, pH 2, and 1.2 g/L AC) (Q_0: mg/g, K_L: L/mg, K_F: L/g, K_T: mg/L and B_1: mg/g)

Adsorbent	Langmuir isotherm				Freundlich isotherm			Temkin isotherm		
	Q_{max}	K_L	R_L	R^2	K_F	$1/n$	R^2	K_T	B_1	R^2
AC	AR14									
	2.50	0.07	0.22	1.00	0.77	0.24	0.99	1.49	0.45	0.99
	AB92									
	0.69	0.02	0.50	0.99	0.0001	6.40	0.97	0.03	20.26	0.93
SMAC	AR14									
	9.17	0.02	0.54	0.80	0.14	1.91	0.93	0.06	17.04	0.87
	AB92									
	11.77	0.10	0.17	0.90	3.26	0.29	1.00	1.34	2.35	0.89

of K_F, $1/n$ and R^2 (correlation coefficient values of all isotherms models) are shown in Table 1.

The Temkin isotherm is given as:

$$q_e = (RT/b)\ \ln(K_T C_e) \tag{5}$$

which can be linearized as:

$$q_e = B_1\ \ln K_T + B_1 \ln C_e \tag{6}$$

where

$$B_1 = RT/b \tag{7}$$

where K_T is the equilibrium binding constant (L/mol) corresponding to the maximum binding energy and constant B_1 is related to the heat of adsorption.

Temkin isotherm contains a factor that explicitly takes into account adsorbing species' adsorbent interactions. This isotherm assumes that the heat of adsorption of all the molecules in the layer decreases linearly with coverage due to adsorbent–adsorbate interactions, and the adsorption is characterized by a uniform distribution of binding energies, up to some maximum binding energy [32,33]. A plot of q_e vs. ln C_e enables the determination of the isotherm constants B_1 and K_T from the slope and the intercept, respectively.

To study the applicability of the Temkin isotherm for the dye adsorption onto AC and SMAC, linear plot q_e vs. ln C_e is plotted. The values of K_T, B_1 and R^2 (correlation coefficient values of all isotherms models) are shown in Table 1.

The correlation coefficient values (R^2) show that the dye removal isotherm using AC and SMAC follows the Langmuir and Freundlich isotherms respectively (Table 1). This means that the adsorption of dyes takes place at specific homogeneous sites and a one layer dye adsorption onto the AC surface. But the dye adsorption onto the SMAC surface takes place at specific heterogeneous sites.

3.6. Adsorption kinetic

The mechanism of solute sorption onto a sorbent can be expressed by several models. To design a fast and effective model, investigations were made on the adsorption rate. For the examination of controlling mechanisms of the adsorption process, such as chemical reaction, diffusion control, and mass transfer, several kinetics models are used to test the experimental data [34,35].

Pseudo-first-order equation is generally represented as follows [36,37].

$$\mathrm{d}q_t/\mathrm{d}t = k_1(q_e - q_t) \tag{8}$$

where q_t and k_1 are the amount of dye adsorbed at time t (mg/g) and the equilibrium rate constant of pseudo-first-order kinetics (1/min), respectively. After integration by applying conditions, $q_t = 0$ at $t = 0$ and $q_t = q_t$ at $t = t$, then Eq. (8) becomes

$$\log(q_e - q_t) = \log(q_e) - (k_1/2.303)t \tag{9}$$

The straight-line plots of $\log(q_e – q_t)$ vs. t for the adsorption of AR14 and AB92 onto AC and SMAC at different adsorbent dosages have also been tested to obtain the rate parameters. The k_1, the experimental q_e ($(q_e)_{\mathrm{Exp.}}$) and correlation coefficients under different dye concentration values were calculated from these plots and are given in Table 2.

Data were applied to the pseudo-second-order kinetic rate equation, which is expressed as [36,38]:

$$\mathrm{d}q_t/\mathrm{d}t = k_2(q_e - q_t)^2 \tag{10}$$

where k_2 is the equilibrium rate constant of pseudo-second order (g/mg min). On integrating Eq. (10),

$$t/q_t = 1/kq_e^2 + (1/q_e)t \tag{11}$$

To understand the applicability of the model, linear plots of t/q_t vs. t for the adsorption of dyes onto AC and SMAC are plotted. The k_2, the experimental q_e ($(q_e)_{\mathrm{Exp.}}$), and correlation coefficients were calculated from these plots and are given in Table 2.

The possibility of intraparticle diffusion resistance affecting adsorption was explored by using the intraparticle diffusion model as [5,39–41]:

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

where k_p is the intraparticle diffusion rate constant.

To understand the applicability of the model, linear plots of q_t vs. $t^{1/2}$ for the adsorption of dyes onto AC and SMAC are plotted. The k_p, I, and correlation coefficients were calculated from these plots and are given in Table 2.

The linear fit between the t/q_t vs. contact time (t) and calculated correlation coefficients (R^2) for a pseudo-second-order kinetic model shows that the dye removal kinetic onto both AC and SMAC can be approximated as pseudo-second-order kinetic (Table 2). In addition, the experimental q_e ($(q_e)_{\mathrm{Exp.}}$) values agree with the calculated ones ($(q_e)_{\mathrm{Cal.}}$) obtained from the linear plots of pseudo-second-order kinetics (Table 2).

Table 2
Kinetic constants for dye adsorption onto AC and SMAC at different adsorbent dosages (200 mL solution, T 25°C, pH 2 and 1.2 g/L AC) ($(q_e)_{Exp.}$: mg/g, $(q_e)_{Cal.}$: mg/g, k_1: 1/min; k_2: g/mg min and k_p: mg/g min$^{1/2}$)

Adsorbent	Dosage (g)	$(q_e)_{Exp.}$	Pseudo-first order			Pseudo-second order			Intraparticle diffusion		
			$(q_e)_{Cal.}$	k_1	R^2	$(q_e)_{Cal.}$	k_2	R^2	k_p	I	R^2
AC	AR14										
	0.300	1.937	1.069	0.051	0.059	1.945	3.108	0.999	0.100	1.391	0.335
	0.600	1.917	1.194	0.219	0.838	1.953	0.627	0.999	0.066	1.497	0.724
	0.900	1.897	0.259	0.115	0.045	1.901	9.540	1.000	0.009	1.847	0.519
	1.200	1.868	0.569	0.113	0.095	1.880	2.301	1.000	0.013	1.842	0.537
	AB92										
	0.300	4.958	1.164	0.071	0.030	4.975	13.467	1.000	0.026	4.814	0.347
	0.600	3.332	1.694	0.150	0.106	3.344	4.064	1.000	0.021	3.215	0.522
	0.900	2.731	1.222	0.562	0.130	2.740	4.037	1.000	0.027	2.581	0.331
	1.200	2.332	1.641	0.177	0.850	2.667	0.226	0.999	0.131	1.762	0.914
SMAC	AR14										
	0.300	13.276	1.884	0.083	0.500	13.889	0.026	0.998	1.039	6.788	0.715
	0.600	8.639	6.486	0.161	0.848	8.929	0.071	0.999	0.475	5.769	0.858
	0.900	6.624	4.721	0.161	0.849	6.849	0.065	0.999	0.377	4.112	0.915
	1.200	5.818	4.613	0.154	0.858	6.024	0.114	0.999	0.309	3.959	0.835
	AB92										
	0.300	9.329	4.335	0.083	0.356	9.804	0.022	0.995	0.533	5.189	0.993
	0.600	8.623	4.920	0.064	0.477	9.174	0.020	0.994	0.731	3.291	0.981
	0.900	7.866	4.295	0.074	0.520	8.264	0.025	0.994	0.583	3.508	0.991
	1.200	7.183	4.111	0.097	0.623	7.463	0.035	0.997	0.496	3.588	0.964

3.7. Desorption studies

The regeneration of the adsorbent is one of the important factors for an economical treatment process. Desorption studies help in the recovery of adsorbate and adsorbent. Dye desorption tests showed that the maximum dye release of 85% for AR14 and 83% for AB92 was achieved in an aqueous solution at pH 12. At high pH value, the number of positively charged sites decreases which favors the desorption of dyes and regeneration of adsorbent.

4. Conclusion

The present paper investigates the surface modification of AC by alkaline (NaOH) and dye removal ability of SMAC. SMAC exhibited higher dye adsorption capacities toward AR14 and AB92 than that of AC. The results showed that the experimental data were correlated reasonably well by Langmuir and Freundlich isotherms for AC and SMAC, respectively. The data indicate that the adsorption kinetics of dyes on AC and SMAC followed the pseudo-second order. Based on the data of present study, one could conclude that the SMAC is an eco-friendly adsorbent for dye removal from colored textile wastewater.

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