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Soy meal hull activated carbon: preparation, characterization and dye adsorption properties

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ABSTRACT

In this paper, the preparation, characterization and dye adsorption properties of soy meal hull activated carbon (SMH-AC) were investigated. Physical characteristics of SMH-AC were studied using Fourier transform infrared (FTIR) and scanning electron microscopy (SEM). Two textile dyes, Acid Red 14 (AR14) and Acid Red 18 (AR18), were used as model compounds. The effects of operational parameter such as SMH-AC dosage, initial dye concentration, pH and salt on dye removal were evaluated. The isotherm of dye adsorption was studied. The data were evaluated for compliance with the Langmuir and Freundlich isotherm models. It was found that AR14 and AR18 followed with Langmuir isotherm. Based on the data of present investigation, one could conclude that the SMH-AC being an eco-friendly and low-cost adsorbent might be a suitable alternative to remove dyes from colored aqueous solutions.

Keywords: Soy meal hull; Activated carbon; Preparation; Characterization; Dye removal; Textile; Wastewater

1. Introduction

Colored effluent arises from the textile companies, dye manufacturing industries, pulp mills, tanneries, electroplating factories, food companies, etc. [1]. Dyes have complex aromatic structures, which make them more stable to chemical and biological degradation [2]. Several methods such as adsorption [3–6], nanofiltration [7], ozonation [8], and electrocoagulation [9] were used to remove dyes from wastewater. Among the wastewater treatment processes, adsorption has been found to be superior to other techniques in terms of ease of operation, simplicity of design, initial cost and insensitivity of toxic substances [10].

Activated carbon is a dye adsorbent with great success because of its high dye removal capacity. The commercial available activated carbons are considered expensive because they are usually derived from wood or coal [2]. There is a need to prepare low cost and effective carbons that can be applied to remove dyes from aqueous phase. The availability and abundance of agricultural waste materials make them good sources of raw materials to prepare the activated carbons [10]. Removal of dyes was studied by several agricultural waste activated carbons [1,2,11–22].

A literature review showed that dye removal using soy meal hull activated carbon (SMH-AC) was not evaluated in details. In this paper, the preparation, characterization and dye adsorption properties of SMH-AC were studied. Two textile dyes (Acid Red 14 (AR14) and Acid Red 18 (AR18)) were used as model compounds. Physical characteristics of SMH-AC were investigated. The isotherm of dye adsorption was studied.

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2. Materials and methods

2.1. Chemicals

Soy meal hull (SMH) was obtained from a local field in north of Iran. Anionic dyes, AR14 and AR18, were used in this study. The dyes were purchased from Ciba Ltd. Dyes were used without further purification. The chemical structure of dyes was shown in Fig. 1. All other chemicals were of analytical grade and obtained from Merck (Germany).

2.2. Preparation of SMH-AC

The SMH was firstly washed to remove the adhering dirt, and then was dried (in a drier for 2 h at 105°C until a constant weight was reached) and crushed. After drying, they were sieved to the required particle size of <0.125 mm. Then SMH was soaked in a nitric acid solution (10 wt.%) for 24 h. The sample is then decanted, dried in an oven at 105°C then carbonized in a muffle furnace for 1 h at 900°C in the absence of air [7]. The activated carbon treated with HNO_3 was obtained.

2.3. Physicochemical characterization of SMH-AC

To determine the isoelectric point (point of zero charge) of SMH-AC, 0.2 g of adsorbent was added to 40 ml of sodium nitrate (0.1 M) at different pH values (2, 3, 4, 5, 6, 7, 8, 9, 10, and 11) and agitated for 24 h at room temperature. The final pH value of solutions was measured [23].

Fourier transform infrared (FTIR) spectra (Perkin-Elmer Spectrophotometer Spectrum One) in the range 450–4000 cm^{-1} were studied. The morphological structure of the SMH-AC was examined by scanning electron microscopy (SEM) using LEO 1455VP scanning microscope.

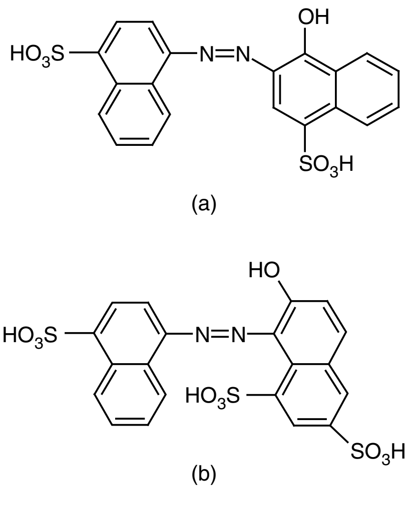

Fig. 1. The chemical structure of dyes (a) AR14 and (b) AR18.

2.4. Adsorption procedure

The dye adsorption measurements were conducted by mixing of SMH-AC for AR14 and AR18 in jars containing 250 ml of a dye solution. The solution pH was adjusted by adding a small amount of H_2SO_4 or NaOH. The change on the absorbance of all solution samples were monitored and determined at certain time intervals during the adsorption process. At the end of the adsorption experiments, the solution samples were centrifuged and the dye concentration was determined. The results were verified with the adsorption isotherms.

UV–VIS spectrophotometer CECIL 2021 was employed for absorbance measurements of samples. The maximum wavelength (λ_{max}) used for determination of residual concentration of AR14 and AR18 at pH 2 in supernatant solution using UV–VIS spectrophotometer were 517 and 509 nm, respectively.

The effect of SMH-AC dosages on dye removal was investigated by contacting 250 ml of dye solution with initial dye concentration of 50 mg l^{-1} and pH = 2 using jar test at room temperature (25°C) for 60 min at a constant stirring speed of 200 rpm. Different amounts of SMH-AC (0.3–0.9 g) were applied to remove AR14 and AR18.

The effect of pH on dye removal was investigated by contacting 250 ml of dye solution with SMH-AC (0.7 g) and initial dye concentration (50 mg l^{-1}) using jar test at room temperature (25°C) for 60 min at a constant stirring speed of 200 rpm. Different pH values (2, 5, 8 and 10) were applied to remove AR14 and AR18.

The effect of initial dye concentration on dye removal was investigated by contacting 250 ml of dye solution with SMH-AC (0.7 g) and pH = 2 using jar test at room temperature (25°C) for 20 min at a constant stirring speed of 200 rpm. Different initial dye concentrations (25, 50, 75 and 100 mg l^{-1}) were applied to remove AR14 and AR18.

The effect of salt (0.02 mM) on dye removal was investigated by contacting 250 ml of dye solution (50 mg l^{-1}) with SMH-AC (0.7 g) and pH = 2 using jar test at room temperature (25°C) for 20 min at a constant stirring speed of 200 rpm. Different salts ($NaHCO_3$, Na_2CO_3 and Na_2SO_4) were used.

3. Results and discussion

3.1. Characterization of SMH-AC

To determine the isoelectric point (point of zero charge) of SMH-AC, adsorbent was added to sodium nitrate solution at different pH values and agitated for 24 h at room temperature. pH_{PZC} (point of zero charge) for SMH-AC is determined as pH 7.0. At pH < 7, an electrostatic attraction exists between anionic dyes and the positively charged surface of the adsorbent due to the ionization of functional groups of adsorbent. As the

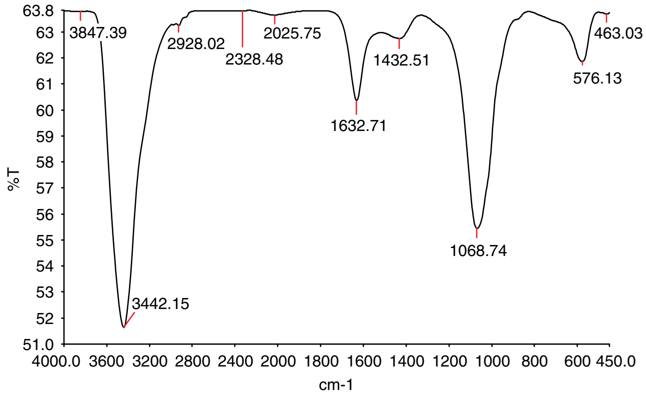

Fig. 2. FT-IR spectrum of SMH-AC.

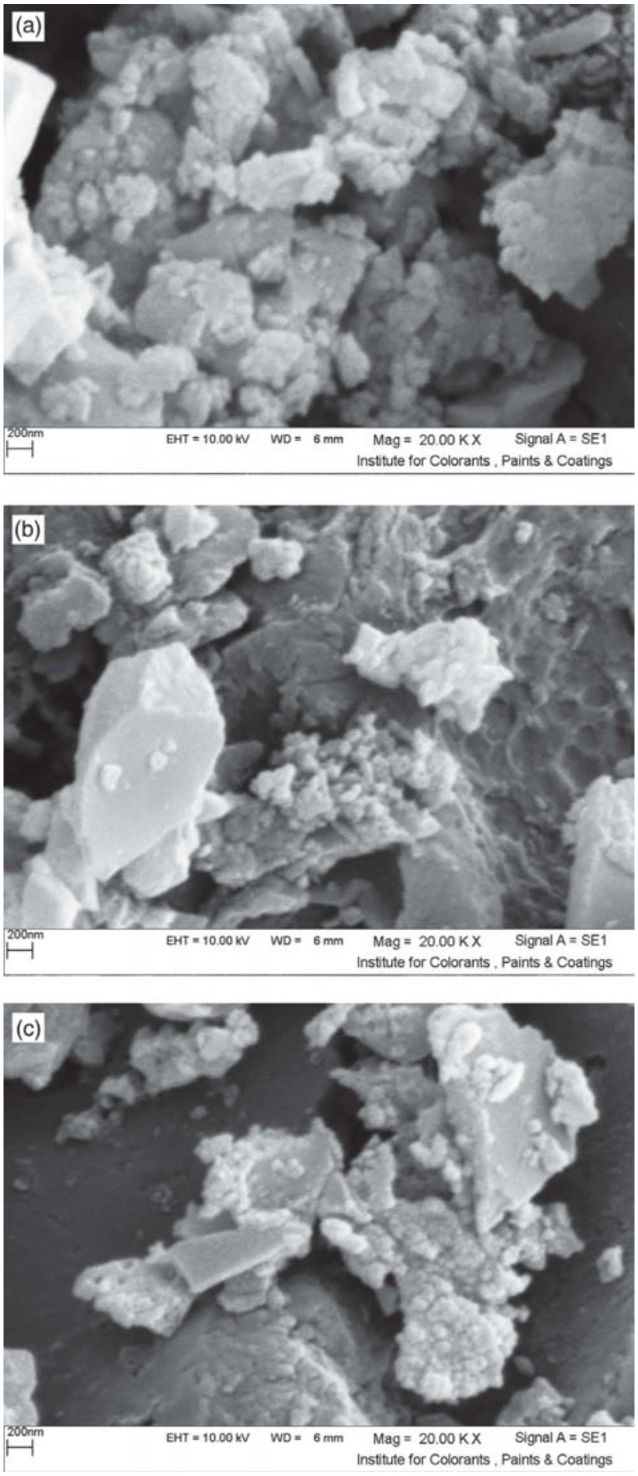

Fig. 3. SEM images (a) SMH-AC, (b) AR14 adsorbed SMH-AC, and (c) AR18 adsorbed SMH-AC.

pH of the system increases, the number of negatively charged sites increases. A negatively charged site on the adsorbent does not favor the adsorption of anionic dyes due to the electrostatic repulsion [23]. Thus pH 2 was used for further experiments.

In order to investigate the surface characteristics of SMH-AC, FTIR in the range 450–4000 cm^{-1} and SEM image were studied. The FTIR spectrum of SMH-AC (Fig. 2) shows that the peak positions are at 3442.15, 2928.02, 1632.71 and 1068.74 cm^{-1}. The band at 3442.15 cm^{-1} is due to O–H stretching. The bands at 2928.02 cm^{-1} correspond to unsymmetrical aliphatic C–H stretching. While the band at 1632.71 cm^{-1} reflects C=C stretching of aromatic rings whose intensity is enhanced by the presence of oxygen atoms as phenol or ether groups. Bands at 1300–1000 cm^{-1} correspond to C–O bending and indicate the presence of phenolic groups [24–27].

SEM has been a primary tool for characterizing the surface morphology and fundamental physical properties of the adsorbent surface. It is useful for determining the particle shape, porosity and appropriate size distribution of the adsorbent. SEM of SMH-AC and dye adsorbed SMH-AC are shown in Fig. 3. From Fig. 3, it is clear that, SMH-AC has considerable numbers of pores, there is a good possibility for dyes to be trapped and adsorbed into these pores. The SEM pictures of SMH-AC samples show very distinguished dark spots which can be taken as a sign for effective adsorption of dye molecules in the cavities and pores of this adsorbent.

3.2. *Effect of operational parameter on dye removal by SMH-AC*

3.2.1. *Effect of adsorbent dosage*

The plot of dye removal (%) versus time (min) at different SMH-AC dosage (g) was shown in Fig. 4.

The increase in dye adsorption with adsorbent dosage can be attributed to increased adsorbent surface and availability of more adsorption sites. However, if the adsorption capacity was expressed in milligrams adsorbed per gram of material, the capacity decreased with the increasing amount of SMH-AC. It can be attributed to overlapping or aggregation of adsorption sites

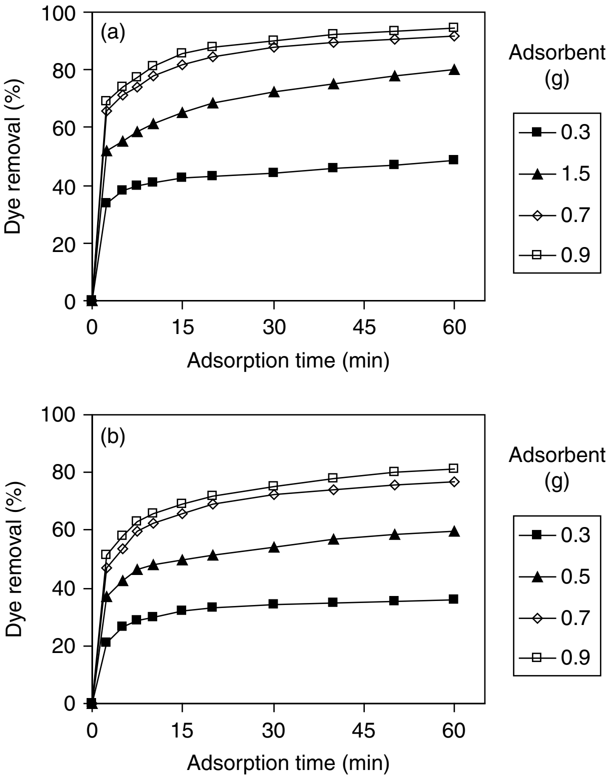

Fig. 4. The effect of adsorbent dosage on dye removal by SMH-AC (a) AR14, (b) AR18 (pH = 2, $T = 25°C$, $C_0 = 50$ mg l^{-1} and 250 ml of solution).

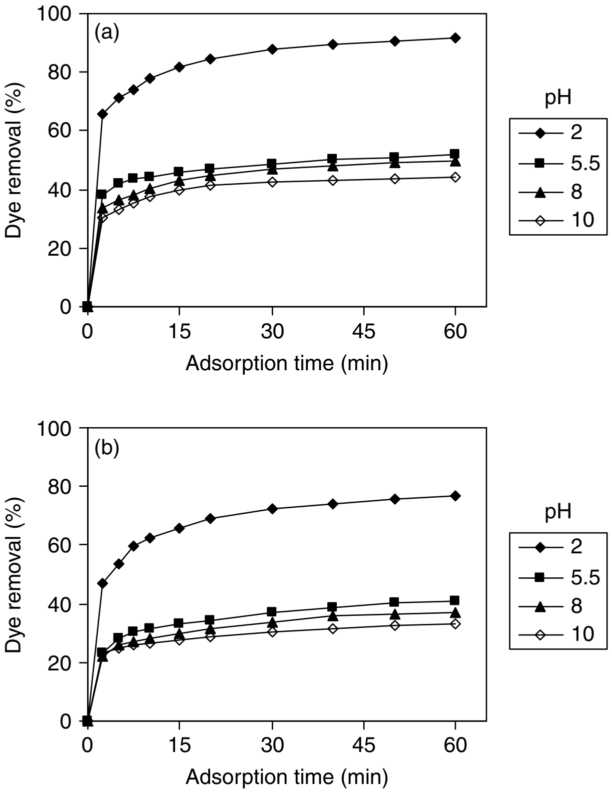

Fig. 5. The effect of pH on the adsorption of dyes on SMH-AC ($C_0 = 50$ mg l^{-1}, $T = 25°C$, $C_{Ad} = 0.7$ g and 250 ml of solution).

resulting in a decrease in total adsorbent surface area available to the dye and an increase in diffusion path length [28].

3.2.2. Effect of pH

The effect of pH on the adsorption of AR14 and AR18 onto SMH-AC is shown in Fig. 5. For two dyes, the adsorption capacity increases when the pH is decreased. Maximum adsorption of anionic dyes occurs at acidic pH (pH 2). At various pH values, the electrostatic attraction as well as the organic property and structure of dye molecules and SMH-AC could play very important roles in dye adsorption on SMH-AC. At pH 2, a significantly high electrostatic attraction exists between the positively charged surface of the adsorbent, due to the ionization of functional groups of adsorbent and negatively charged anionic dye. As the pH of the system increases, the number of negatively charged sites is increased. A negatively charged site on the adsorbent does not favor the adsorption of anionic dyes due to the electrostatic repulsion [29]. Also, lower adsorption of AR14 and AR18 dyes at alkaline pH is due to the presence of excess OH^- ions destabilizing anionic dyes and competing with the dye anions for the adsorption sites. The effective pH was 2 and it was used in further studies.

3.2.3. Effect of dye concentration

Adsorption can generally be defined as the accumulation of material at the interface between two phases [30]. The adsorption efficiencies of AR14 and AR18 on SMH-AC were evaluated. The influence of varying the initial dye concentration of two dyes was assessed. The results are shown in Fig. 6. It is obvious that the higher the initial dye concentration, the lower the percentage of dye adsorbed.

The dye adsorbed onto SMH-AC increases with an increase in the initial dye concentration if the amount of adsorbent is kept unchanged due to the increase in the driving force of the concentration gradient with the higher initial dye concentration. The adsorption of dye by SMH-AC is very intense and reaches equilibrium very quickly at low initial concentration. At a fixed SMH-AC dosage, the amount of dye adsorbed increased with increasing concentration of solution, but the percentage of adsorption decreased. In other words, the residual dye concentration will be higher for higher initial dye concentrations. In the case of lower concentrations, the ratio of initial number of dye moles to the available adsorption sites is low and subsequently the fractional adsorption becomes independent of initial concentration [31–34].

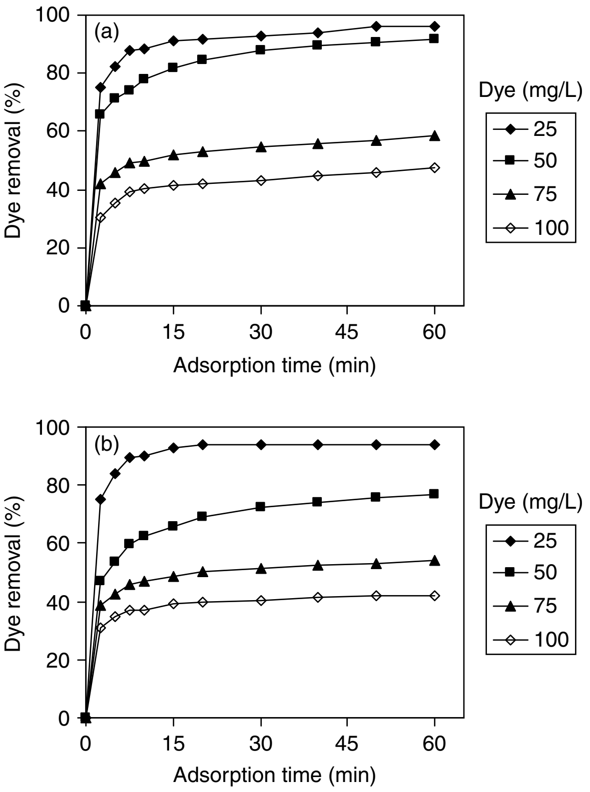

Fig. 6. The effect of dye concentration on dye removal by SMH-AC (a) AR14, (b) AR18 (pH = 2, T = 25°C, C_{Ad} = 0.7 g and 250 ml of solution).

3.2.4. Inorganic salts effect

The occurrence of dissolved inorganic ions is rather common in dye-containing industrial wastewater [35]. These substances may compete for the active sites on the adsorbent surface or deactivate the adsorbent and, subsequently, decrease the dye adsorption efficiency. A major drawback resulting from the high reactivity and non-selectivity of adsorbent is that it also reacts with non-target compounds present in the background water matrix, that is, dye auxiliaries present in the exhausted reactive dye bath. It results higher adsorbent dosage demand to accomplish the desired degree of dye removal efficiency.

To investigate inorganic salts effect on dye removal efficiency, 0.02 mM of $NaHCO_3$, Na_2CO_3 and Na_2SO_4 were used. Fig. 7 illustrates that dye removal capacity of SMH-AC is decreased in the presence of inorganic salts. The ionic strength increases as NaCl concentration increase and more Na^+ and Cl^- ions may screen the electrostatic interaction of opposite charges in material and dye molecules, leading to the decrease of the amount adsorbed (and also the adsorption rate).

3.3. Adsorption isotherm

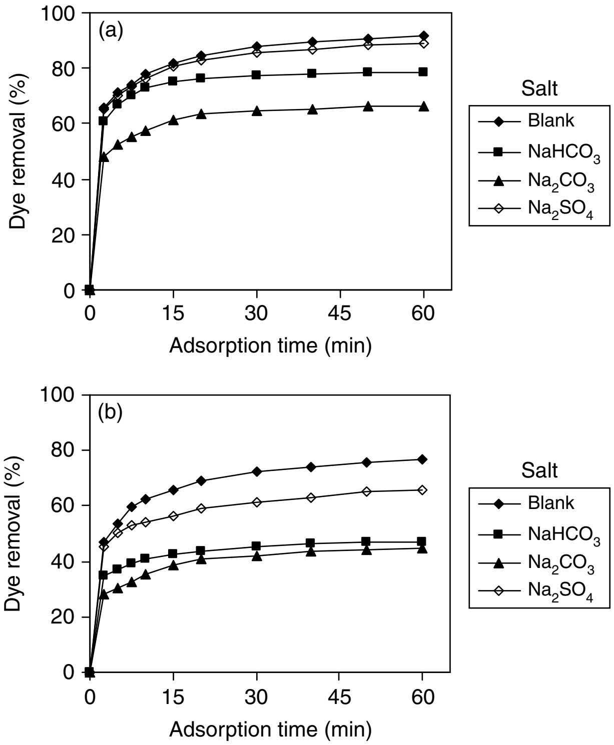

Fig. 7. The effect of salt on the adsorption of dyes on SMH-AC (salt: 0.02 mM, C_0 = 50 mg l^{-1}, pH = 2, T = 25°C, C_{Ad} = 0.7 g and 250 ml of solution).

The adsorption isotherm expresses the relation between the mass of the dye adsorbed at a particular temperature, the pH, particle size and liquid phase of the dye concentration.

The Langmuir isotherm which has been successfully applied to many adsorption processes can be used to explain the adsorption of dye into adsorbent. A basic assumption of the Langmuir theory is that adsorption takes place at specific sites within the adsorbent [1,36–38]. The Langmuir equation can be written as follows:

$$q_e = \frac{Q_0 K_L C_e}{1 + K_L C_e} \tag{1}$$

where q_e, C_e, K_L and Q_0 are the amount of dye adsorbed on SMH-AC at equilibrium (mg g^{-1}), the Langmuir concentration of dye solution (mg l^{-1}), equilibrium constant (l g^{-1}) and the maximum adsorption capacity (mg g^{-1}), respectively.

The linear form of Langmuir equation is:

$$\frac{C_e}{q_e} = \frac{1}{K_L Q_0} + \frac{C_e}{Q_0} \tag{2}$$

Also, Isotherm data were tested with Freundlich isotherm that can be expressed by [1,39]:

$$q_e = K_F C_e^{1/n} \tag{3}$$

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

$$\log q_e = \log K_F + (1/n)\log C_e \tag{4}$$

The Temkin isotherm is given as:

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

which can be linearized as:

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

where:

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

Temkin isotherm contains a factor that explicitly takes into the account adsorbing species adsorbent interactions. This isotherm assumes that (1) the heat of adsorption of all the molecules in the layer decreases linearly with coverage due to adsorbent–adsorbate interactions, and that (2) the adsorption is characterized by a uniform distribution of binding energies, up to some maximum binding energy [40,41]. A plot of q_e versus ln C_e enables the determination of the isotherm constants B_1 and K_T from the slope and the intercept, respectively. K_T is the equilibrium binding constant (l mol^{-1}) corresponding to the maximum binding energy and constant B_1 is related to the heat of adsorption.

To study the applicability of the Langmuir, Freundlich and Temkin isotherms for the dye adsorption onto SMH-AC at different adsorbent dosage and dye concentrations, linear plots of C_e/q_e against C_e, log q_e versus log C_e and q_e versus ln C_e are plotted and shown in Figs. 8–10, respectively. The values of Q_0, K_L, K_F, $1/n$, 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 SMH-AC does not follow the Freundlich and Temkin isotherms (Table 1). The linear

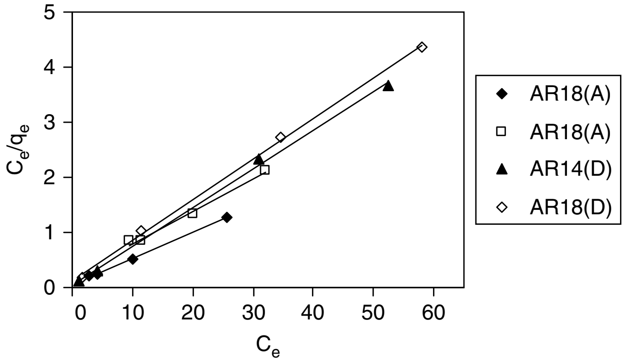

Fig. 8. Langmuir adsorption isotherms of dye adsorption onto SMH-AC at different adsorbent dosages (A) and dye concentrations (D).

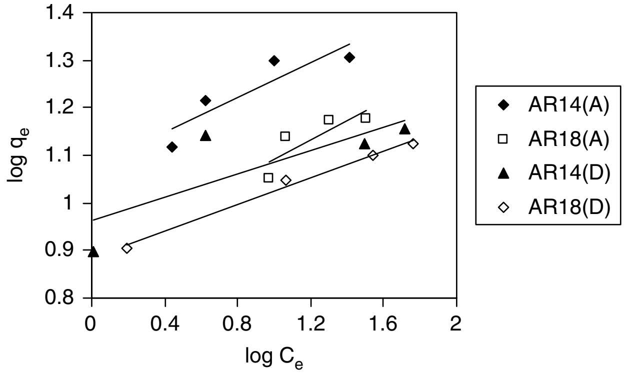

Fig. 9. Freundlich adsorption isotherms of dye adsorption onto SMH-AC at different adsorbent dosages (A) and dye concentrations (D).

Table 1
Linearized isotherm coefficients for dye adsorption onto SMH-AC at different adsorbent dosages and dye concentrations

	Langmuir			Freundlich			Temkin		
	Q_0	K_L	R^2	K_F	$1/n$	R^2	K_T	B_1	R^2
At different adsorbent dosages (g)	AR14								
	21.367	0.771	0.998	11.890	0.183	0.801	38.993	3.074	0.826
	AR18								
	16.835	0.290	0.990	3.764	0.202	0.693	10.338	2.679	0.717
At different dye concentrations (mg l^{-1})	AR14								
	17.007	1.046	0.998	10.109	0.144	0.649	422.563	1.744	0.641
	AR18								
	13.624	0.519	0.999	7.686	0.140	0.991	171.727	1.453	0.998

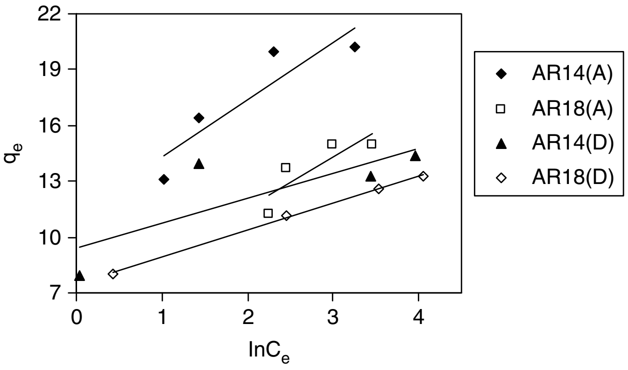

Fig. 10. Temkin adsorption isotherms of dye adsorption onto SMH-AC at different adsorbent dosages (A) and dye concentrations (D).

fit between the C_e/q_e versus C_e and calculated correlation coefficients (R^2) for Langmuir isotherm model show that the dye removal isotherm can be approximated as Langmuir model (Table 1). This means that the adsorption of dyes takes place at specific homogeneous sites and a one layer adsorption onto SMH-AC surface.

4. Conclusions

The preparation, characterization and dye adsorption properties of SMH-AC were investigated. SMH-AC was studied using FTIR and SEM. Equilibrium studies were done for the adsorption of AR14 and AR18 from aqueous solutions onto SMH-AC. The equilibrium data showed that data for AR14 and AR18 followed with Langmuir isotherm. The results showed that the SMH-AC being an eco-friendly and low-cost adsorbent with relatively large adsorption capacity might be a suitable alternative to remove dyes from colored wastewater.

Acknowledgments

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