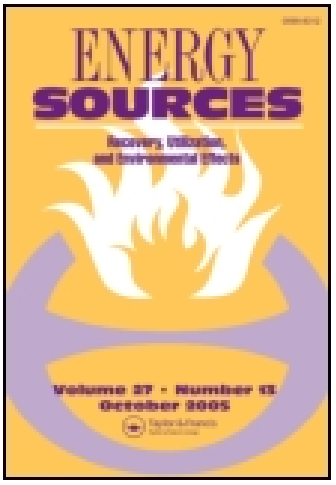

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Removal of Methylene Blue from Aqueous Media by Using Cokes Obtained from Lignite Pyrolysis

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Effect of the initial dye concentration, contact time, pH, adsorbent quantity, and temperature on the adsorption of the cokes was investigated for methylene blue adsorption of the cokes obtained from pyrolysis of Mustafa Kemal Pasa lignite. The maximum amount of dye adsorbed was 270 mg/g. Equilibrium of the adsorption was examined by Langmuir and Freundlich isotherms. The pseudo-first-order, pseudo-second-order, and intra-particle diffusion models described the adsorption kinetics. The adsorption equilibrium was well described by Langmuir and Freundlich isotherms. The adsorption kinetics fit well to the Ho's pseudo-second-order model. The cokes are highly favorable adsorbents for the removal of methylene blue from aqueous media.

Keywords: adsorption, cokes, kinetics, methylene blue removal, thermodynamics

INTRODUCTION

There are over 100,000 synthetic dye materials in current industrial usage with an annual consumption rate of nearly 700,000 tons (Meyer, 1981; Zollinger, 1987; Christie, 2007). A vast amount of industrial effluents is discharged into natural water resources with complex environmental and health consequences on multiple levels: toxic effects on living organisms (Walsh et al., 1980; Brown and De Vito, 1993; Bae and Freeman, 2007), discolorification, and decreased transparency of natural water resources (Christie, 2007; Nemerrow, 1978; Rai et al., 2005). Various techniques have been developed to extract and eliminate these materials from the colored effluents, including biological treatment, coagulation, adsorption, oxidation, filtration, and electrochemical methods. However, many of these techniques are highly specialized to treat specific types of materials, and are excessively costly or difficult to implement, limiting their usage by the industry. Among the most promising techniques, biological treatment methods suffer because living organisms as adsorbent are unable to disintegrate certain dye materials (Gupta and Suhas, 2009). Chemical treatment methods are more powerful, but most of them introduce other toxic materials and/or have an unavoidable sludge problem (Muthuraman et al., 2009). The most popular adsorbent for adsorption is granular active carbon but it requires regeneration to vacant its active sites prior to reuse, which is a complex and expensive process. Therefore, there is much recent activity to develop or identify alternative absorbent materials, which have comparable effectiveness at a lower cost. To this end, several low-cost materials have been investigated, such as pomegranate

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peel (Amin, 2009), silkworm pupa (Noroozi et al., 2007), hazelnut shells (Ferrero, 2007), carbon nanotubes (Wu, 2007), wheat bran carbon (Özer and Dursun, 2006), calcium alginate beads (Aravindhan et al. 2006), wheat shells (Bulut and Aydın, 2005), etc.

Adsorption characteristics of Turkish lignites have been investigated by many Turkish researchers so far. However, most of these studies were focused on determination of adsorption properties of lignites and coals. This work was aimed to present the adsorption properties of the cokes obtained from the lignites since cokes have better surface characteristics for the adsorption properties, such as increased pore volume and surface area.

MATERIALS AND METHODS

Cokes used as absorbents in this study were obtained from the pyrolysis of Mustafa Kemal Pasa (MKP) lignite at four different temperatures (500, 600, 800, and 900°C). The details of this process were given in (Bozkurt et al. 2008). Zinc-free Methylene Blue (MB) (C.I. no. 52015) (Merck) was used as the cationic dye material.

A series of experiments were conducted to investigate the effect of initial dye concentration, amount of adsorbent, pH level, agitation speed, temperature, and contact time. For each of these experiments, 100 mL of aqueous dye solution was prepared varying only one parameter and fixing the others. Agitation of the mixtures was achieved by using an Incubator (Clifton NE5-28) and the pH values of the solutions were measured by a pH meter (PH523, WTW). Afterwards, the remaining cokes were seperated with a centrifuge (Hettich EBA III), and the concentration in the supernatant dye solution was analyzed with a UV-visible spectrometer system (Cary 50–Bio/Varian) by monitoring the change in absorption at 660 nm, which was found to be the peak absorption wavelength in the *pre-phase* experiments.

RESULTS AND DISCUSSION

The amount of dye adsorbed at equilibrium, q_e (mg/g), is calculated according to the equation below:

$$q_e = \frac{(C_0 - C_e)V}{m}, \tag{1}$$

where V is the volume of the solution, C_0 (mg/L) and C_e (mg/L) are the initial and equilibrium concentrations of the adsorbate, and m (g) is the amount of adsorbent.

Effect of the Initial Dye Concentration

A general trend of increasing adsorption with increasing initial dye concentration can be seen from Figure 1a), which is a well-known phenomenon for fixed volume of solution and adsorbent quantity; at initial adsorption stages, there is a large number of vacant sites on the adsorbent surface. In addition, there is mass-transfer resistance between solute concentrations of dye in the solution and on the surface of adsorbent for MB molecules to migrate. A higher number of MB molecules increases the driving force for the diffusion process and alters the mass-transfer resistance, resulting in an increase in the amount of adsorbed MB molecules. Similar observations have been found in previous studies (Aravindhan et al., 2006; Bulut and Aydın, 2005; Bozkurt et al., 2008; Han et al., 2006; Mane et al., 2007). It can be seen from Figure 1a that maximum adsorption value, $q_{\max}$, is 96 mg/g for MKP 500°C coke with an initial MB concentration of 12

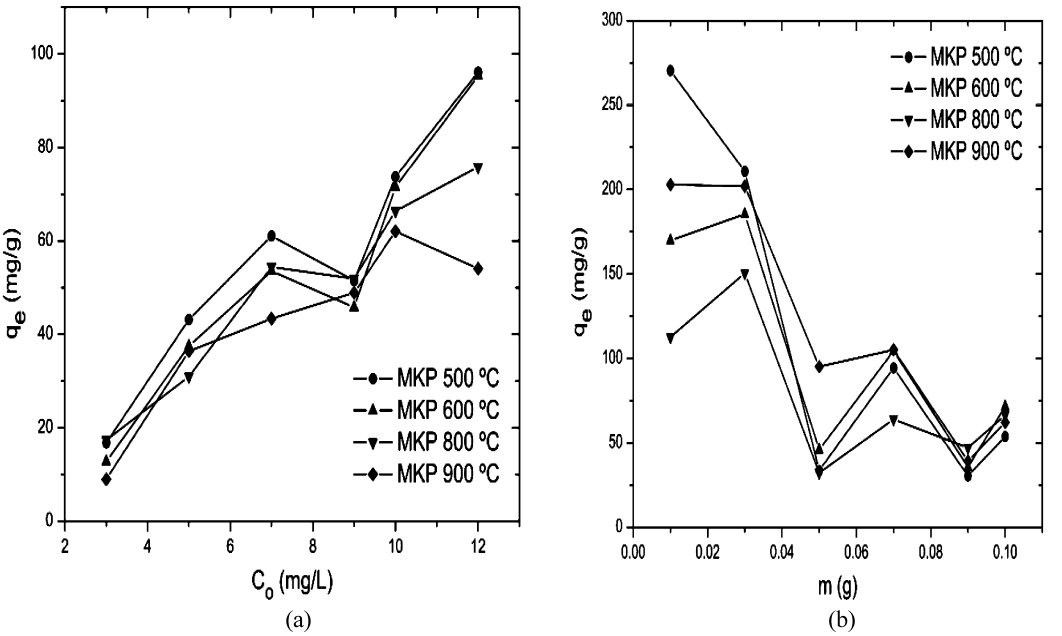

FIGURE 1 (a) Effect of initial dye concentration on the adsorption of MB onto MKP coke ($T = 30°C$, $m = 0.1$ g, $V = 100$ mL, 400 rpm, $t = 50$ min, and pH = 7.2). (b) Effect of adsorbent quantity on the adsorption of dye MB onto MKP coke ($C_o = 10$ mg/L, $T = 30°C$, $V = 100$ mL, 400 rpm, $t = 50$ min, and pH = 7.2).

mg/L. Similarly, q_{max} values for MKP 600, 800, and 900°C cokes are found to be 95, 76, and 62 mg/g, respectively.

Effect of the Adsorbent Quantity

The q_e values decrease with increasing adsorbent quantity (Figure 1b) in accordance with Eq. (1), which predicts an inverse relationship between q_e and m for fixed volume of solution and initial dye concentration. Increasing adsorbent quantity increases the concentration gradient between the solute concentrations of dye in the solution and on the surface of adsorbent, leading to reduced q_e values for unit quantity of adsorbent. These findings are in agreement with previous studies (Kumar and Kumaran, 2005; Royer et al., 2009). Simiarly, q_{max} is found to be 270 mg/g for MKP 500°C coke with 0.01 g of adsorbent. For MKP 600, 800, and 900°C cokes, q_{max} values are 185, 150, and 203 mg/g, respectively.

Effect of the Agitation Speed

It can be seen from Figure 2a that the q_e values tend to decrease when agitation speed increases from 100 to 200 rpm. Further increase to 400 rpm leads to an increase in q_e values again. Agitation during adsorption increases the mobility of MB molecules and adsorbent particles, decreasing the resistivity of the boundary layer and causing a small change in concentrations between aqueous and solid phases (Al-Ghouti et al., 2009). A sudden decrease in adsorption at 200 rpm can be understood as follows: At this agitation speed, the system has already reached equilibrium by formation of a stable boundary layer between the aforementioned phases, and the

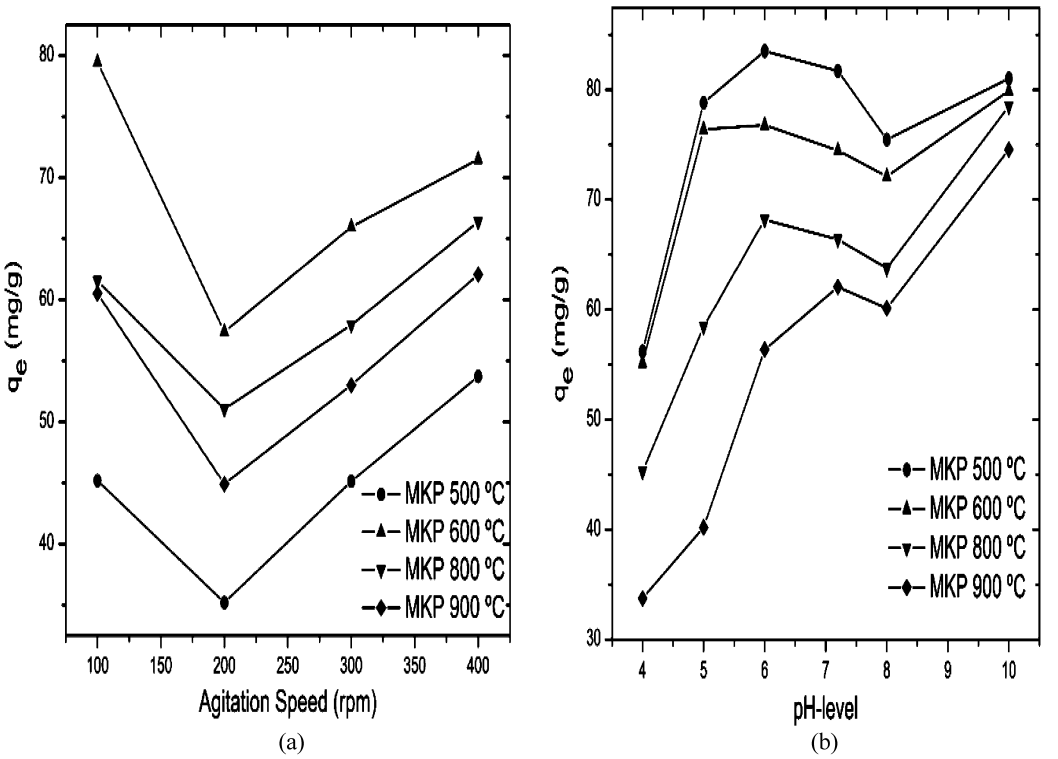

FIGURE 2 (a) Effect of agitation speed on the adsorption of dye MB onto MKP coke (Co = 10 mg/L, m = 0.1 g, T = 30°C, V = 100 mL, t = 50 min, and pH = 7.2). (b) Effect of pH level on the adsorption of dye MB onto MKP coke (C_o = 10 mg/L, m = 0.1 g, T = 30°C, V = 100 mL, t = 50 min, and 400 rpm).

adsorption cannot increase further. Increasing the agitation speed further to 400 rpm disrupts the stable boundary layer and leads to an increased adsorption again. Similar observations have been reported previously (Gürses et al., 2005). The maximum adsorption value, q_{max}, is determined to be 71 mg/g for MKP 600°C coke at an agitation speed of 400 rpm. For MKP 500, 800, and 900°C cokes, the corresponding q_{max} values are 53 and 62 mg/g, respectively.

Effect of the pH Level

It has been found that adsorption (q_e) increases with pH level, over the range of 4 to 10 (Figure 2b). Under strongly acidic conditions, the adsorbent surface gets positively charged and MB cations cannot be adsorbed efficiently as a result of competition between MB cations and H+ ions. The increase in adsorption may be attributed to the vast amount of available binding sites on the adsorbent surface. Adsorption for neutral pH levels remains nearly constant. Under strongly basic conditions, the adsorption tends to increase due to negative charging of the adsorbent surface and electrostatic attractions between the adsorbent surface and MB cations. These results are consistent with Bulut and Aydın (2005) and Gürses et al. (2005). At a fixed pH level of 7.2, maximum adsorption levels, q_{max}, are found to be 81, 79, 78, and 74 mg/g for MKP 500, 600, 800, and 900°C cokes.

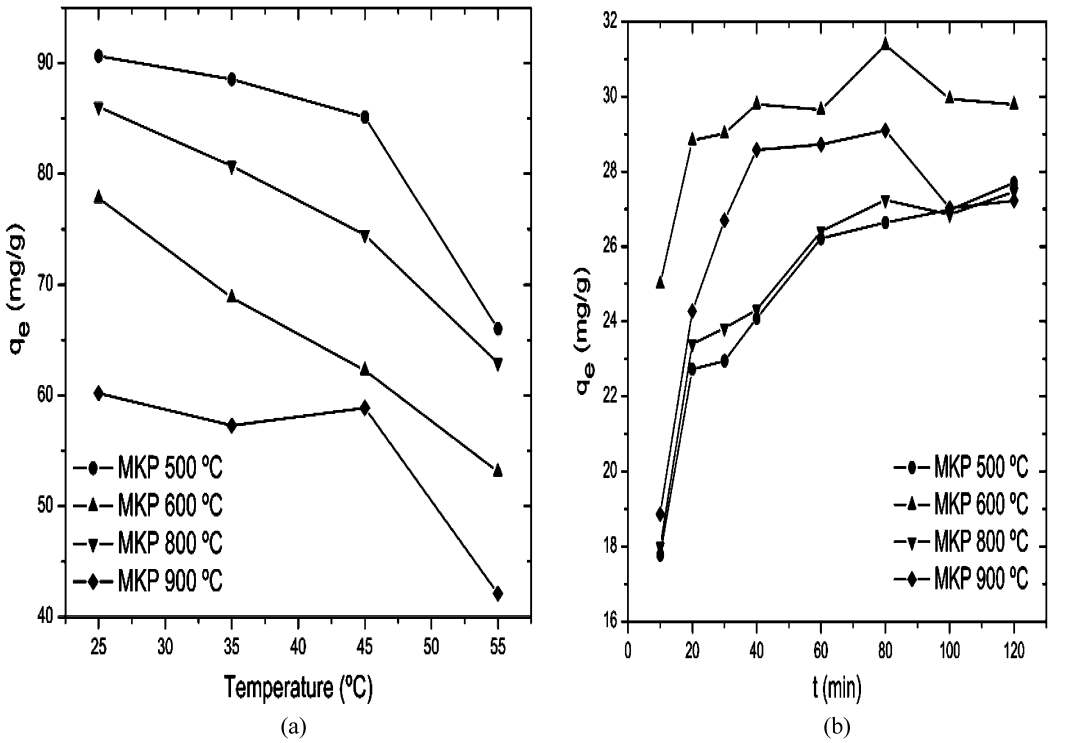

FIGURE 3 (a) Effect of temperature on the adsorption of dye MB onto MKP coke ($Co = 10$ mg/L, $m = 0.1$ g, $V = 100$ mL, 400 rpm, $t = 50$ min, and pH = 7.2). (b) Effect of contact time on the adsorption of dye MB onto MKP coke ($C_o = 10$ mg/L, $m = 0.1$ g, $T = 30°C$, $V = 100$ mL, 400 rpm, and pH = 7.2).

Effect of Temperature on Adsorption

The effect of temperature on adsorption is summarized in Figure 3a, which shows that the *qe* values decrease with increasing temperature. This is to be expected since the adsorption process is exothermic. The decrease may be influenced by the increasing solubility of MB ions in aqueous media with increasing temperature. In addition, a possible increase in solubility may weaken the interaction between adsorbate and adsorbent, resulting in a decrease in adsorption (Iqbal and Ashiq, 2006). Maximum adsorption value for MKP 500°C coke is found to be 88 mg/g at 25°C. q_{max} values for MKP 600, 800, and 900°C cokes are found to be 77, 86, and 60, respectively.

Effect of Contact Time

The amount of adsorption, q_e, increases along with contact time (Figure 3b). For the first 20 min, there is rapid adsorption of dye and, thereafter, the adsorption rate decreases gradually, and adsorption reaches equilibrium in approximately 50 min. The initial rapid increase in adsorption is due to the vast amount of available vacant sites in the initial stages of adsorption. Following rapid occupation of the vacancies by MB ions, the concentration gradient between adsorbate in solution and adsorbate in the adsorbent surface reduces along with the adsorption rate. Similar results have been reported in Kumar and Kumaran (2005) and Vadivelan and Kumar (2005). It was found that the maximum adsorption value was 31 mg/g for MKP 600°C coke in 80 min. At MKP 500, 800, and 900°C, q_{max} values were 28, 27, and 29 mg/g, respectively.

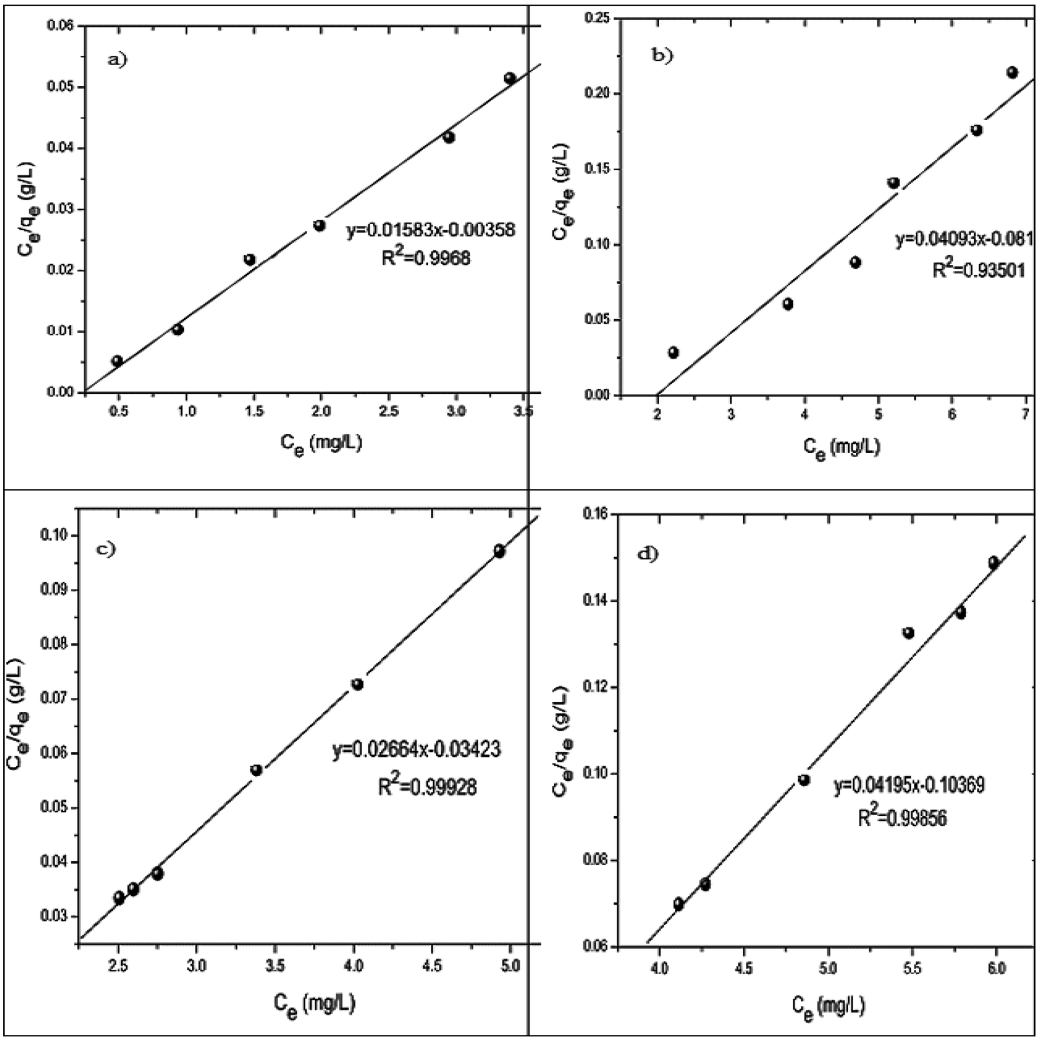

FIGURE 4 Langmuir isotherm plots of the adsorption of MB onto: (a) MKP 500°C, (b) MKP 600°C, (c) MKP 800°C, and (d) MKP 900°C coke.

Adsorption Equilibrium

In this study, the adsorption equilibrium has been expressed by Langmuir and Freundlich isotherms. The linearized Langmuir equation is given by Langmuir (1918):

$$\frac{C_e}{q_e} = \frac{1}{bq_{\max}} + \frac{C_e}{q_{\max}}, \tag{2}$$

where b is a Langmuir constant at equilibrium related to the identity of binding sites (L/mg), C_e represents the dye concentration at equilibrium, and $q_{\max}$ is an indication of adsorption capacity when the surface is fully covered. $q_{\max}$ and b can be calculated from the slope and intercept of

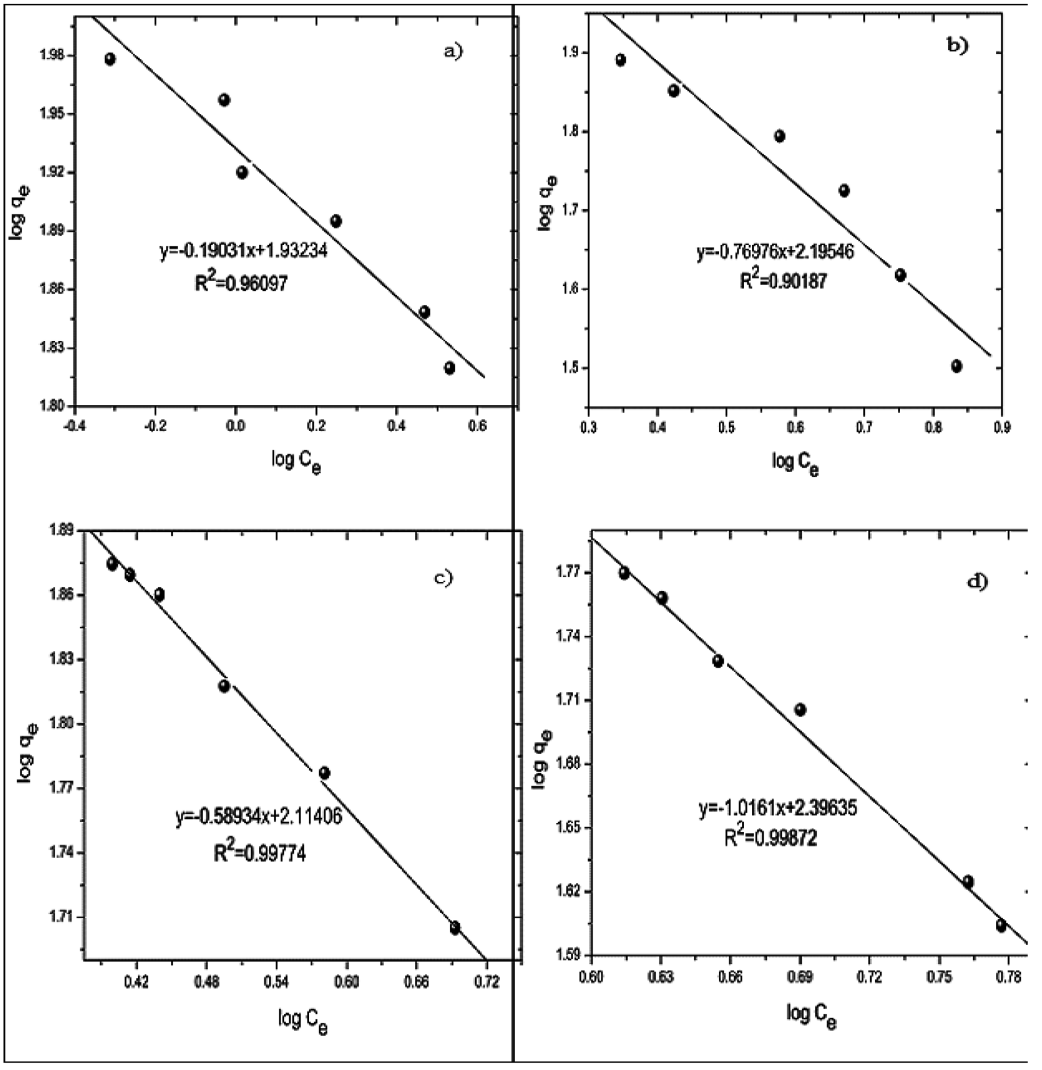

FIGURE 5 Freundlich isotherm plots of the adsorption of MB onto: (a) MKP 500°C, (b) MKP 600°C, (c) MKP 800°C, and (d) MKP 900°C coke.

the straight lines of the plot C_e/q_e versus C_e. In addition, the essential features of the Langmuir isotherm can be expressed in terms of a dimensionless separation factor (R_L), which is defined by the following equation (Weber and Chakkravorti, 1974):

$$R_L = \frac{1}{1 + (bC_0)}, \tag{3}$$

for $R_L > 1$, adsorption is unfavorable, $R_L = 1$ adsorption is linear; for $0 < R_L < 1$, adsorption is favorable, and $R_L = 0$ adsorption is irreversible.

TABLE 1
Langmuir and Freundlich Isotherm Coefficients for the Adsorption of MB onto MKP Coke at Elevated Temperatures

		Langmuir Isotherm Constants				*Freundlich Isotherm Constants*	
Coke Type	*Langmuir Isotherm* R^2	*b, L/mg*	q_{max}, *mg/g*	R_L	*Langmuir Isotherm* R^2	K_F, *mg/g (L/mg)$^{1/n}$*	*n*
MKP 500°C	0.998	2.598	58.858	0.040	0.973	91.281	3.921
MKP 600°C	0.986	0.874	41.408	0.129	0.969	118.470	1.996
MKP 800°C	0.992	1.644	53.648	0.065	0.964	97.870	3.124
MKP 900°C	0.998	0.422	24.931	0.3106	0.998	232.595	1.029
Adsorbent/Reference	*Langmuir Isotherm* R^2	*b, L/mg*	q_{max}, *mg/g*	R_L	*Langmuir Isotherm* R^2	K_F, *mg/g (L/mg)$^{1/n}$*	*n*
Silkworm pupa (Noroozi et al., 2007)	0.904	0.011	555.5	—	0.946	16.15	1.586
Hazelnut shell (Ferrero, 2007)	0.999	0.36	76.9	0.003	—	—	—
Carbon nanotubes (Wu, 2007)	0.992	0.93	39.84	0.05	0.973	16.26	3.43
Dehydrated wheat bran carbon (Özer and Dursun, 2006)	0.999	0.425	185.2	—	0.999	96.6	13
Calcium alginade beads (Aravindhan et al., 2006)	0.933	0.02	78.125	—	0.999	6.15	1.339
Wheat shells (Bulut and Aydın, 2005)	0.99	0.025	16.56	0.8	0.840	1.46	2.74
Mango seed kernel powder (Kumar and Kumaran, 2005)	0.999	0.197	142.857	—	0.916	43.904	3.501

The Freundlich isotherm is a reasonably accurate empirical isotherm that can be used to describe heterogeneous systems, and the linearized Freundlich equation is given by Freundlich (1906):

$$\log q_e = \log K_F + \left(\frac{1}{n}\right) \log C_e, \tag{4}$$

where K_F[(mg/g) (L/mg)$^{1/n}$] is related to the adsorption capacity and ($1/n$) represents the adsorption intensity. These constants can be determined from the linear plot of log q_e versus log C_e.

The linear plots of the Langmuir and Freundlich isotherms for MKP 500, 600, 800, and 900°C cokes are shown in Figures 4 and 5, respectively. The isotherm data was calculated using the least-square method and the related correlation coefficients (R^2 values) and isotherm constants for four types of MKP coke are given in Table 1. The high R^2 values demonstrate that both the Langmuir and the Freundlich equations are good fits for the adsorption process. However, the Langmuir isotherm fits to the experimental data better than Freundlich isotherm, which suggests a homogeneous distribution of active sites on the coke surface. $q_{\max}$ values calculated for the Langmuir isotherm (Table 1) range from 24 to 59 mg/g.

Comparison of various parameters characterizing the potential utility of the MKP cokes and other low-cost adsorbents for the adsorption of MB from aqueous media was given in Table 1. The $q_{\max}$, K_F, and b values of MKP cokes compare very favorably. R_L values range between 0 and 1, contributing to a favorable adsorption.

TABLE 2
Pseudo-first-order and Ho's Pseudo-second-order Kinetic Models and Intra-particle Diffusion Model Coefficients for the Adsorption of MB onto MKP Coke at Different Time Intervals

	Pseudo-first-order Model Constants				*Pseudo-second-order Model Constants*			*Intraparticle Diffusion Model Constants*		
Coke Type	R_1^2	k_1, min^{-1}	q_e (calc.), mg/g	q_e (exp.), mg/g	R_2^2	k^2 g/mg.min	q_e, mg/g	R_{id}^2	K_p, $mg/g.min^{1/2}$	B, mg/g
MKP 500°C	0.962	0.028	11.516	28	0.999	0.005	29.053	0.871	1.111	16.460
MKP 600°C	0.340	0.010	4.162	32	0.998	0.027	30.479	0.537	0.494	25.648
MKP 800°C	0.908	0.244	8.891	28	0.999	0.006	28.720	0.806	1.029	17.347
MKP 900°C	0.232	0.010	4.828	30	0.995	0.020	28.193	0.472	0.846	2.768
Adsorbent/Reference	R_1^2	k_1, min^{-1}	q_e (calc.), mg/g	q_e (exp.), mg/g	R_2^2	k^2 g/mg.min	q_e, mg/g	R_{id}^2	K_p, $mg/g.min^{1/2}$	B, mg/g
Hazelnut shell (Ferrero, 2007)	0.999	0.264	24.7	25	1	0.0390	25	—	—	—
Carbon nanotubes (Wu, 2007)	0.976	0.0130	23.56	27.2	0.990	0.00316	27.20	0.893	0.2456	4.48
Dehydrated wheat bran carbon (Özer and Dursun, 2006)	0.976	0.0018	48.20	66.50	0.997	0.00010	69.90	—	—	—
Calcium alginade beads (Aravindhan et al., 2006)	0.957	0.0138	1.222	5.460	0.999	0.0387	5.5142	0.884	0.0729	—
Wheat shells (Bulut and Aydın, 2005)	0.9641	0.035	16.34	13.70	0.997	0.0040	17.92	0.953	0.054	10.10

The applicability of the pseudo-first-order model, Ho's pseudo-second-order equation, and intraparticle diffusion models are analyzed for the adsorption of MB onto MKP cokes. The best-fit models are determined based on the calculated linear regression correlation coefficient, R^2. The calculated kinetic constants are shown in Table 2. The authors note that correlation coefficients are highest for Ho's pseudo-second-order equation, which suggests that this model is best suited to predicting the kinetics of the MB adsorption onto MKP cokes. Assuming there is an exchange of electrons between MB molecules and adsorbent through covalent forces, the adsorption process can be described as chemisorption (Iqbal and Ashiq, 2006; Vadivelan and Kumar, 2005), although the thermodynamic studies strongly suggest that the adsorption process is physisorption.

CONCLUSIONS

MKP cokes are effective adsorbents of MB from aqueous media. The results of this study indicated that the best performance was obtained with the 500°C coke. Adsorption capacity of these cokes was found to be proportional to the initial dye concentration, contact time, and pH level, and inversely proportional to adsorbent quantity and temperature. However, the Langmuir isotherm fits the experimental data better than Freundlich isotherm, which indicates a homogenous distribution of binding sites on the adsorbent surface. High b values of Langmuir isotherm confirm the high affinity of the binding sites on the adsorbent surface. The kinetic studies show that the rate-limiting step of the adsorption of MB onto cokes is the external diffusion. The adsorption is characterized as physisorption and it is reversible.

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