Full Length Research Paper

Isothermal, kinetic and thermodynamic studies on basic dye sorption onto tartaric acid esterified wheat straw

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In this paper, a new cation sorbent, which bore carboxyl and hydroxyl groups of tartaric acid (TA) derived from esterified wheat straw (EWS), was originally prepared by solid phase thermochemistry method. The isotherm, kinetics and thermodynamics of basic dye sorptions from aqueous solution onto EWS were investigated for the first time. Two basic dyes, methylene blue (MB) and crystal violet (CV) were selected as sorbates. The isothermal data correlated with the Langmuir model better than the Freundlich model. The maximum sorption capacity (Q_m) of EWS for MB and CV was 129.87 and 112.36 mg/g, respectively. The equilibriums of dye sorptions were respectively reached about 13 and 18 h for MB and CV. The sorption processes could be described by the pseudo-second-order kinetic model and there were two intra-particle diffusion steps in the dye sorption processes. The thermodynamic study indicated that the dye sorptions were spontaneous and endothermic in nature.

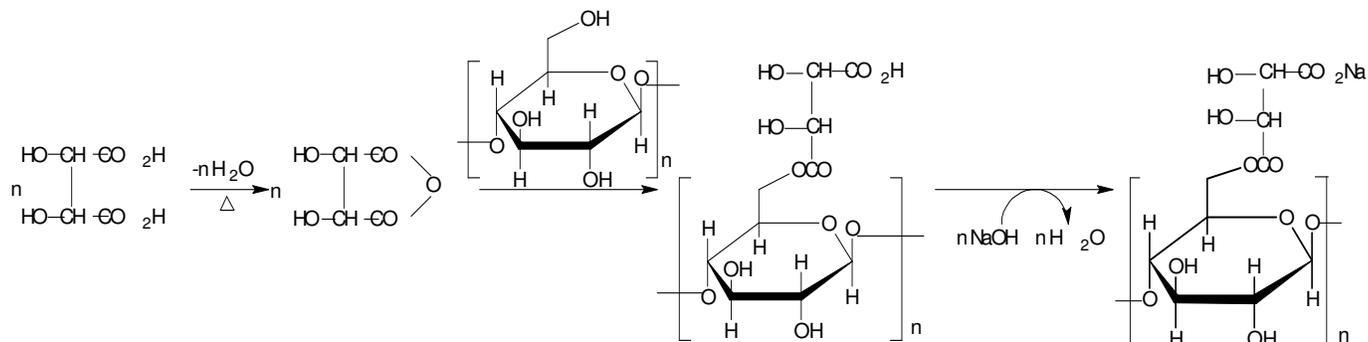**Key words:** Isotherm, kinetics, thermodynamics, basic dye, sorption.

INTRODUCTION

Dyes are synthetic aromatic compounds which can bring bright and firm color to other materials. They are also important water pollutants which are generally present in the effluents of textile, paper, rubber, plastic, leather, cosmetic, food and drug industries. It is reported that over 100,000 commercially available dyes exist and more than 7×10^5 metric tones of dyes are produced worldwide annually (Pearce et al., 2003; McMullan et al., 2001). Dye wastewater discharged by various industries causes' environmental hazards. From the environmental view-point, the removal of dyes from wastewater is a great mission, since some dyes or their metabolites may be toxic as well as carcinogenic, mutagenic and teratogenic (McKay et al., 1985; Gregory et al., 1991). Some biological and physical/chemical methods have been employed for dye wastewater treatment. In all these methods, the sorption has been found to be economical and effective dye wastewater treatment technique as it can remove various dyes with lower treatment cost.

At present, there is a growing interest in using low cost sorbents for dye sorption. If a sorbent is inexpensive and ready for use, the sorption process will be a promising technique. Recently, many lignocellulosic materials had been used as low cost sorbent for removal of dyes from water, which included banana pith (Namasivayam et al., 1993), coir pith (Namasivayam and Kadirvelu, 1994), palm-fruit bunch (Nassar and Magdy, 1997), eucalyptus bark (Morais et al., 1999), apple pomace and wheat straw (Robinson et al., 2002), kudzu (Allen et al., 2003), rice husk (Vadivelan and Kumar, 2005), pine sawdust (Ozacar and Sengil, 2005), peanut hull (Gong et al., 2005), mango seed kernel (Kumar and Kumaran, 2005), lemon peel (Kumar, 2007), kohlrabi peel (Gong et al., 2007), palm kernel fibre (Ofomaja, 2007), almond shell (Ardejani et al., 2008), castor seed shell (Oladoja et al., 2008), coffee husk (Oliveira et al., 2008), neem leaf powder (Tahir et al., 2008) and luffa fiber (Demir et al., 2008). Generally, dye sorption capacity of natural lignocellulosic materials is low. For improving their dye sorption capacity, some lignocellulosic materials had been subjected to chemical modification (Low and Lee, 1997; Marchetti et al., 2000; Gong et al., 2006).

This work originally described the solid phase preparation

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Figure 1. The thermochemical esterification mechanism of wheat straw.

method of thermochemically esterified wheat straw with tartaric acid (TA) to produce a low-cost cation sorbent. The sorption isotherm, kinetics and thermodynamics of two basic dyes, methylene blue (MB) and crystal violet (CV), sorbed onto esterified wheat straw (EWS) from aqueous solutions were investigated. As we know, it was the first time that the kinetics and thermodynamics of dye sorption on TA esterifying lignocellulosic materials were studied. The purpose of the present work was to exploit and utilize new low cost sorbent for enhancing the treatment of environmental pollution and reducing the cost of pollution treatment.

MATERIALS AND METHODS

Preparation of EWS

Wheat straw, supplied by a local farm, was cut into pieces of 10 cm length and washed with tap water to remove soil and dust and then dried overnight at 50°C. Dried wheat straw segments were ground and sieved to retain the 20 - 40 mesh fractions for further chemical modification.

The chemical modification of wheat straw was carried out according to the following method: Ground wheat straw was mixed with 0.5 mol/dm^3 TA at the ratio of 1:12 (straw: acid, w/v) and stirred for 30 min. The acid/straw slurry was placed in a stainless steel tray and dried at 50°C in a forced air oven for 24 h. Then, the thermochemical reaction between acid and wheat straw was performed at 120°C for 90 min. After cooled down to the room temperature, the EWS was extensively washed to neutral pH with distilled water and filtered. The filter residue was suspended in 0.1 mol/dm^3 NaOH at a suitable ratio and stirred for 60 min, followed by washing thoroughly with distilled water to remove residual alkali and then the wet EWS was dried at 50°C until the constant weight and preserved in a desiccator for the further use as sorbent. The thermochemical esterification mechanism of wheat straw can be supposed as shown in Figure 1.

FTIR spectrum and swelling of sorbent

The FTIR spectra of crude wheat straw and EWS were shown in Figure 2. Comparing with the IR spectrum of crude rice straw, it could been seen that there was a obvious characteristic stretching vibration absorption band of carboxyl groups at 1750 cm^{-1} (indicated by arrow) in IR spectrum of EWS. This result confirmed the occurrence of esterifying reaction.

The swelling of sorbent in aqueous solution was considered. The amount of water absorbed by crude wheat straw and EWS was quantitatively determined by absorption equilibrium experiment to be 495 and 580%, respectively. The hydrophilic groups of EWS were more than these of crude wheat straw; so, the swelling of EWS was more than that of crude wheat straw.

Preparation of basic dye solution

Two basic dyes (MB and CV), in commercial purity, were used without further purification. MB and CV are basic dye of phenothiazine type (C.I. No 52,015, FW = 373.88, λ_{max} = 670 nm) and basic dye of triphenylmethane type (C.I. No 42,555, FW = 407.99, λ_{max} = 584 nm), respectively. The dye stock solutions were prepared by dissolving accurately weighted dyes in distilled water to the concentration of 500 mg/dm^3. The experimental solutions were obtained by diluting the dye stock solutions in accurate proportions to different initial concentrations.

Experimental methods and measurements

Except for the thermodynamic study, which was carried out at 293.15, 303.15, 313.15 and 323.15 K using 3 g/dm^3 of EWS dosage for 400 mg/dm^3 of dye solutions, other sorption experiments were conducted in a rotary shaker at 150 rpm and ambient temperature using 250 cm^3 shaking flasks containing 100 cm^3 different concentrations or initial pH values of dye solutions. The initial pH values of the dye solutions were previously adjusted with dilute HNO$_3$ or NaOH using a pH meter. Different doses of sorbent were added to each flask, one flask with dye solution but no sorbent was used as control and then the flasks were sealed up to prevent change of volume of the solutions during the experiments. After shaking the flasks for predetermined time intervals at selected temperatures, the samples were withdrawn from the flasks and the dye solutions were separated from the sorbent by filtration/centrifugation. Dye concentrations in the supernatant solutions were determined by measuring absorbance values at maximum wavelengths of dyes with a 752 W Grating Spectrophotometer (Shanghai, China) and computing from the calibration curve. The amount of dyes sorbed on sorbent was calculated by the mass balance equation. Each run of the experiments was replicated at least three times.

RESULTS AND DISCUSSION

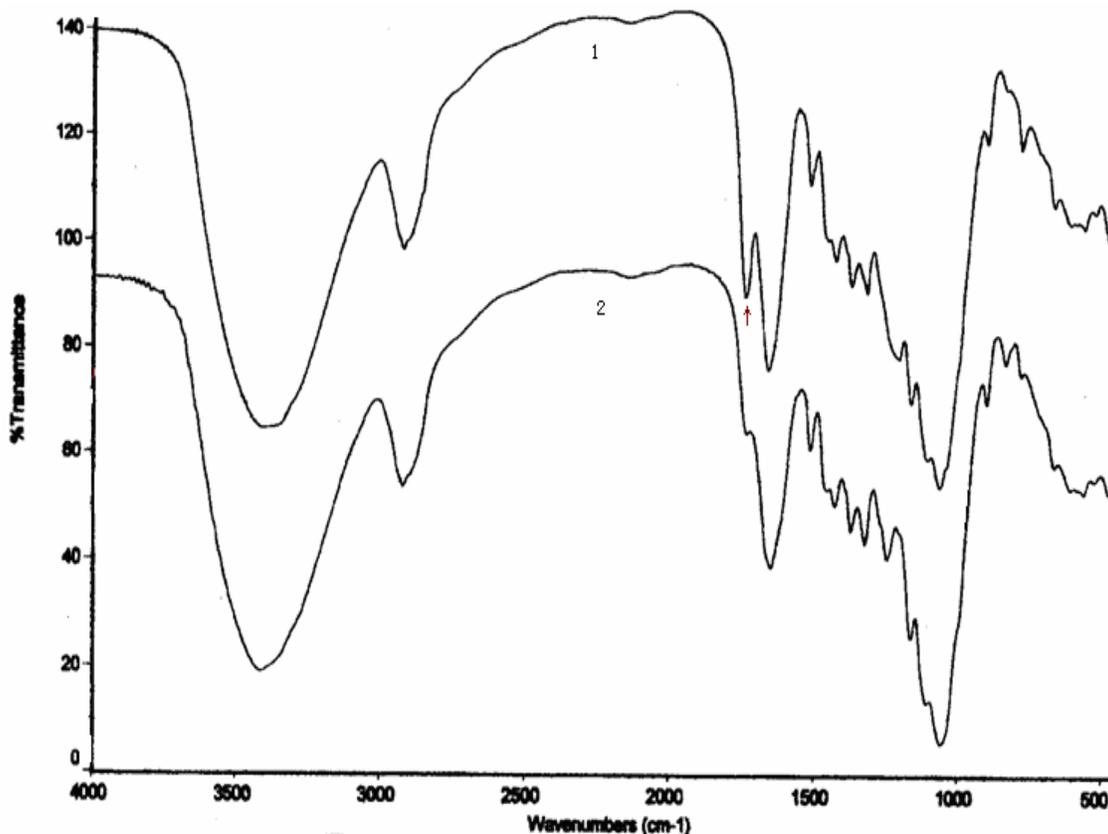The preliminary experiments indicated, beyond pH 4, the

Figure 2. The FTIR spectra of wheat straw (1: EWS; 2: crude wheat straw).

maximum dye removal ratios were reached and the percentages of dyes sorbed kept basically unchangeable. For this reason, the natural pH values of MB and CV solutions were selected as the optimum pH value for the other experiments. For 250 mg/dm^3 of dye solutions, the maximum dye removal percentages were reached by the 3.0 g/dm^3 or up of EWS. So, the EWS dosage of 3.0 g/dm^3 was chosen for the further experiments.

The ion exchange mechanism could be used to explain the experimental result of initial pH influence. The practical functional group of EWS is carboxyl group. At acidic pH, carboxyl group on EWS was protonated into the non ionic form; the non ionic form of carboxyl groups did not favor the sorption of dye cations. As the initial pH was increased, the number of negatively charged carboxyl groups on EWS increased. It favored the sorption of dye cations due to the electrostatic attraction.

Sorption isotherm

The relationships between the initial dye concentrations and sorption percentages of dyes were shown in Figure 3. When the dye concentrations were increased from 50 to 500 mg/dm^3, the percentages of MB and CV sorbed decreased from 98.38 to 73.71% and from 98.01 to

64.09%, respectively.

With the data in Figure 3, Langmuir and Freundlich equations were employed to study the sorption isotherm of MB and CV. The Langmuir equation is based on the assumption that maximum sorption corresponds to saturated monolayer of sorbate molecule on the sorbent surface, that the energy of sorption is constant and that there is no transmigration of sorbate in the plane of the surface. The Langmuir equation was shown as follows:

$$q_e = aQ_mC_e/(1 + aC_e)$$

where C_e (mg/dm^3) is the concentration of the dye solution at equilibrium, q_e (mg/g) is the amount of dye sorbed at equilibrium, Q_m is the maximum sorption capacity and represents a practical limiting sorption capacity when the sorbent surface is fully covered with monolayer sorbate molecules and a is Langmuir constant. Above equation can be linearized in following reciprocal form:

$$C_e/q_e = (1/aQ_m) + (C_e/Q_m)$$

The Q_m and a values could be obtained from the slope (1/Q_m) and intercept (1/aQ_m) of linear plot of C_e/q_e versus C_e.

The empirical Freundlich equation was expressed as
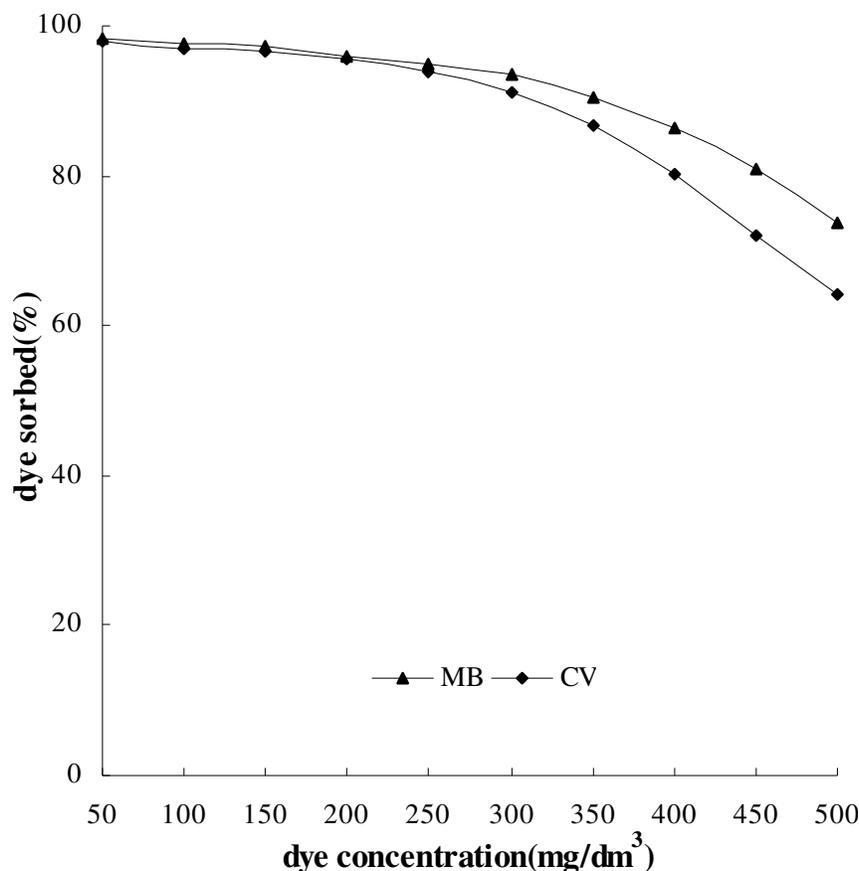
Figure 3. Influence of dye concentration on sorption of MB and CV by EWS (EWS dose: 3g/dm^3; contact time: 20 h; pH: natural).

Table 1. The Langmuir and Freundlich equations, values of constants and correlation coefficients of MB and CV sorbed on EWS.

Dye	Langmuir			Freundlich		
	$Ce/qe = 0.0077 Ce + 0.0555$ (MB)			ln Qe = 0.3867ln Ce + 3.1999 (MB)		
	$Ce/qe = 0.0089 Ce + 0.055$ (CV)			ln Qe = 0.3475ln Ce + 3.1653 (CV)		
	Q_m(mg/g)	a	R^2	K	1/n	R^2
MB	129.87	0.1387	0.9996	24.53	0.3867	0.9197
CV	112.36	0.1618	0.9994	23.70	0.3475	0.8752

follows:

$$Q_e = K C_e^{1/n}$$

where Q_e is the amount of dye sorbed at equilibrium, C_e is the concentration of the dye solution at equilibrium, above equation can be linearized in following logarithmic form:

$$\ln Q_e = \ln K + (1/n) \ln C_e$$

K and $1/n$ are empirical constants and their values could

be calculated from the intercepts (ln K) and slopes (1/n) of linear plots of ln Q_e versus ln C_e.

The Langmuir and Freundlich equations, values of constants and correlation coefficients of MB and CV sorbed on EWS were given in Table 1. Comparing the correlation coefficients in Table 1, it could be found that the sorption isotherms of MB and CV sorbed on EWS followed the Langmuir model better than the Freundlich model. The maximum sorption capacity (Q_m) of EWS for MB and CV was 129.87 and 112.36 mg/g, respectively. The sorption capacities (Q_m) of some sorbents for MB and CV were compared in Table 2.

Table 2. The sorption capacities (Q_m) of some sorbents for MB and CV (ambient temperature).

Dye	Sorbent	Q_m (mg/g)	Reference
MB	EWS	129.87	present study
	dehydrated peanut hull	108.6	Ozer et al., 2007
	wheat straw	63.72	present study
	luffa fiber	49	Demir et al., 2008
	rice husk	40.6	Vadivelan and Kumar, 2005
CV	charred saw dust	195.7	Chakraborty et al., 2005
	EWS	112.36	present study
	wheat straw	54.87	present study
	jute fiber carbon	27.99	Porkodi and Kumar, 2007
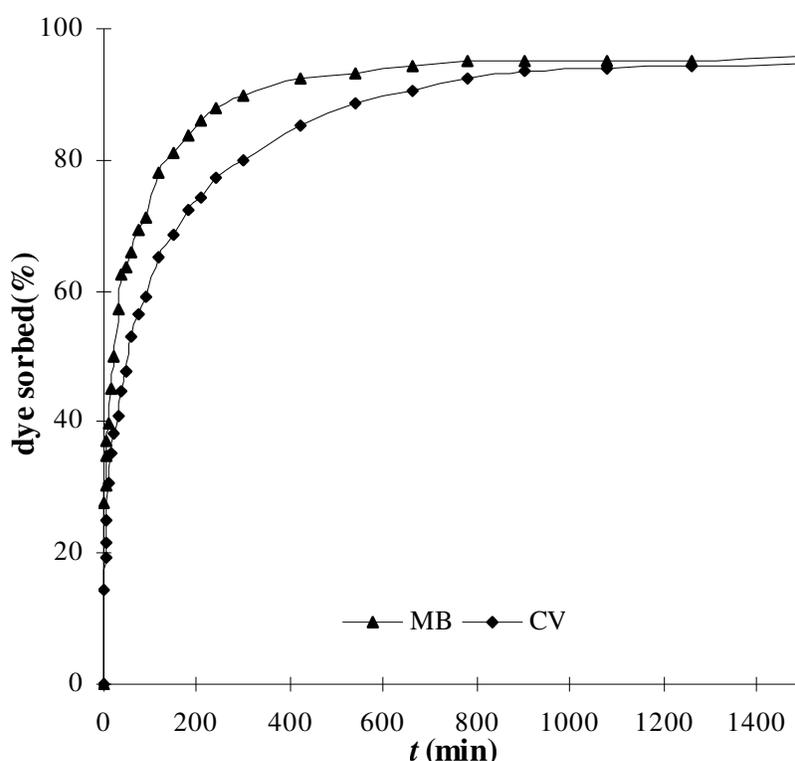
Figure 4. Effect of contact time on sorption of MB and CV by EWS (dye concentration: 250mg/dm^3; EWS dose: 3g/dm^3; pH: natural).

Sorption kinetics

The effects of the contact time on the sorption percen-tages of dyes were presented in Figure 4. The removal rates of dyes were very rapid at the initial stages of sorption. It was caused by the fast diffusion and sorption of dye molecules onto the macropore surface of sorbent. After a very rapid sorption, dye uptake rates slowly de-clined with lapse of time and reached equilibrium values at about 13 and 18 h for MB and CV, respectively. This process was controlled by the micropore diffusion

velocities of dyes into the intraparticle matrix of sorbent.

The data in Figure 4 were treated with the Lagergren's pseudo-first-order rate (Lagergren, 1898) and Ho's pseudo-second-order rate equation (Ho and McKay, 2000). The Lagergren's pseudo-first-order rate equation is expressed as:

$$\ln(q_e-q_t) = \ln q_e - k_{ad} t$$

The Ho's pseudo-second-order rate equation is described as:
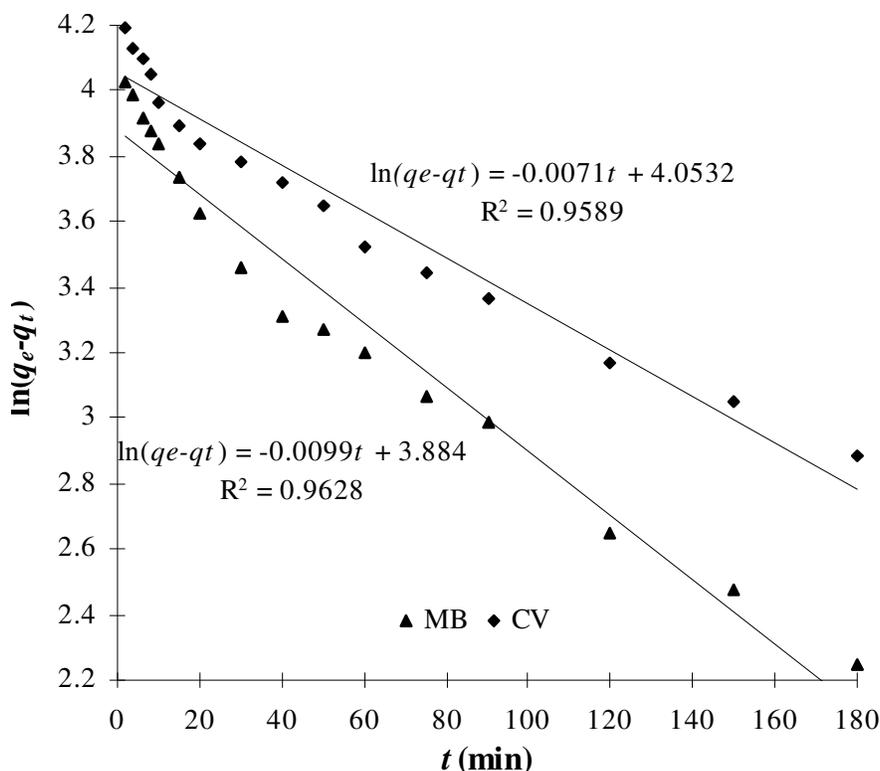
Figure 5. The pseudo-first-order model plots of MB and CV sorbed by EWS.

$t/q_t = (1/k_{ad} q_e^2) + (t/q_e)$

where q_e and q_t (mg/g) refer to the amount of dye sorbed at equilibrium and time t (min), respectively and k_{ad} is the rate constant.

The pseudo-first-order and pseudo-second-order model plots of MB and CV sorbed on EWS were shown in Figures 5 and 6, respectively. Comparing the correlation coefficients in Figures 5 and 6, it could be detected that the kinetics of MB and CV sorbed on EWS conformed to the Ho's pseudo-second-order rate equation better than the Lagergren's pseudo-first-order rate equation.

The data in Figure 4 were also treated with the following intraparticle diffusion rate equation:

$q_t = k_{id} t^{1/2} + C$

where q_t (mg/g) is the amount of dye sorbed at time t (min), k_{id} is the intraparticle diffusion rate constant and C is the intercept. When the line passes through the origin ($C = 0$), the intraparticle diffusion will be the sole rate control step. If the plot does not pass through the origin, it implies that intraparticle diffusion is not the only rate control step but also other processes may control the rate of sorption.

The linear intraparticle diffusion rate plots of MB and CV sorbed on EWS were shown in Figure 7. It could be seen that the plots of q_t versus $t^{1/2}$ exhibited two linear sections with different slopes. The dual linear plots indicated that two intraparticle diffusion steps occurred in the dye sorption processes. The first straight portion was attributed to the macropore diffusion and the second linear portion to micropore diffusion. The similar dual linear plots were also reported in the literature (Goswami and Ghosh, 2005). The fact that the plots did not pass through the origin indicated that although intraparticle diffusion was involved in the dye sorption process, it was not the only rate control step.

Sorption thermodynamics

The thermodynamic parameters, ΔH° and ΔS°, were obtained from the following Van't Hoff equation:

$\ln (q_e/C_e) = (\Delta S^\circ/R) - (\Delta H^\circ/RT)$

where q_e/C_e is the equilibrium constant (cm^3/g), ΔS° is standard entropy (J/mol·K), ΔH° is standard enthalpy (J/mol), T is the absolute temperature (K) and R is the gas constant (8.314 J/mol·K). From Figure 8, the ΔH° and ΔS° values could be calculated from the slope ($-\Delta H^\circ/R$) and intercept ($\Delta S^\circ/R$) of the linear plot of ln (q_e/C_e) versus 1/T.

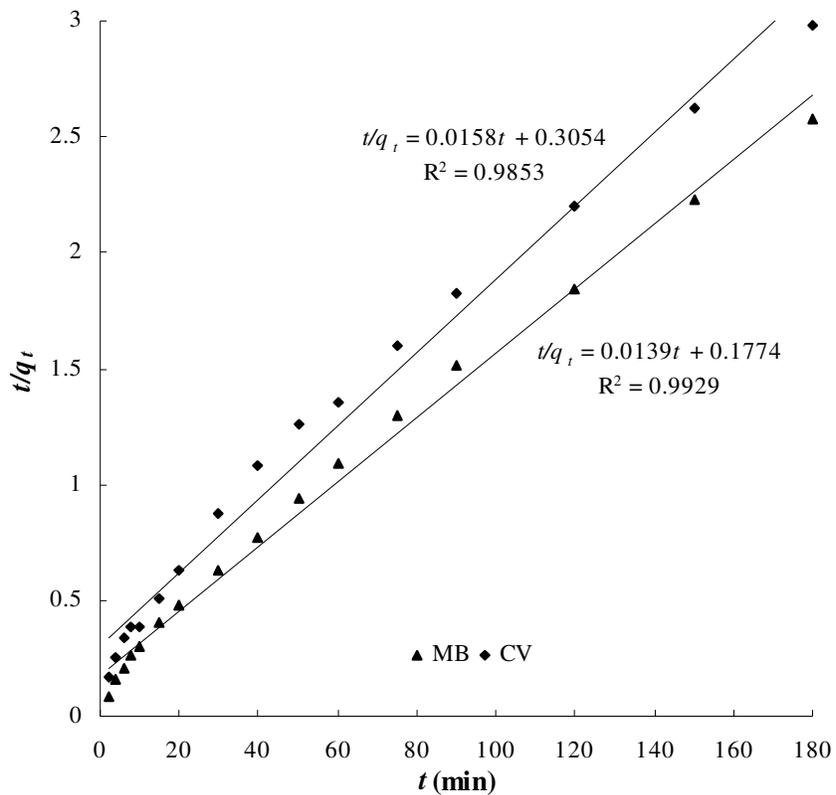The thermodynamic parameter, ΔG°, was calculated

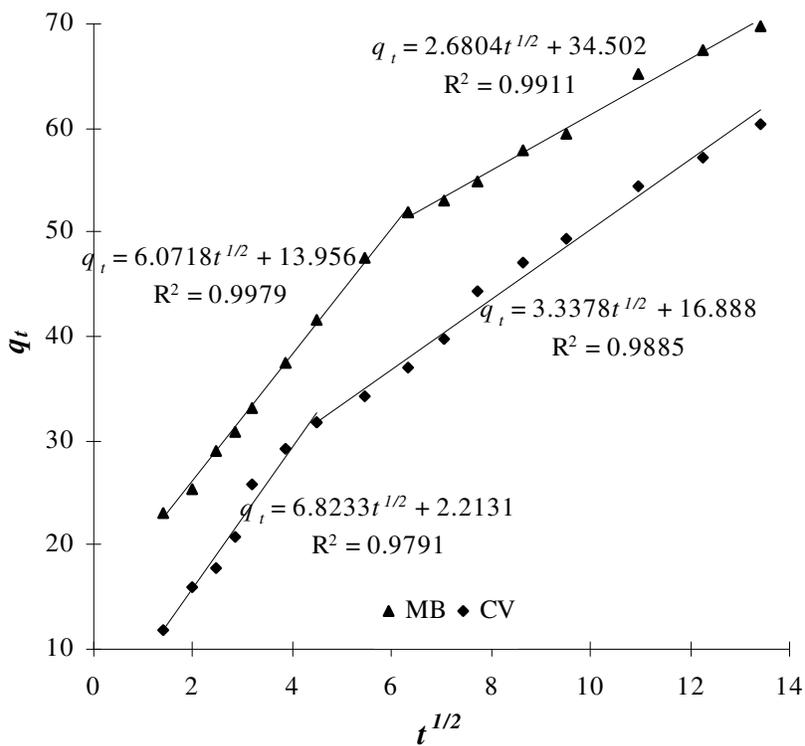**Figure 6.** The pseudo-second-order model plots of MB and CV sorbed by EWS.

$t/q_t = 0.0158t + 0.3054$
$R^2 = 0.9853$

$t/q_t = 0.0139t + 0.1774$
$R^2 = 0.9929$

$q_t = 2.6804t^{1/2} + 34.502$
$R^2 = 0.9911$

$q_t = 6.0718t^{1/2} + 13.956$
$R^2 = 0.9979$

$q_t = 3.3378t^{1/2} + 16.888$
$R^2 = 0.9885$

$q_t = 6.8233t^{1/2} + 2.2131$
$R^2 = 0.9791$

Figure 7. The plot of q_t vs. $t^{1/2}$ for the sorption of MB and CV by EWS.

Table 3. The thermodynamic parameters of some sorbents for MB and CV (323.15 K).

Dye	Sorbent	$\Delta H°$ (kJ/mol)	$\Delta S°$ (J/mol·K)	$\Delta G°$ (kJ/mol)	Reference
MB	EWS	23.23	139.13	- 21.73	present study
	coffee husk	17.69	- 33.1	- 7.39	Oliveira et al., 2008
	dehydrated peanut hull	20.05	155	- 30.12	Ozer et al., 2007
	wheat shell	33.41	185	- 26.35	Bulut and Aydın, 2006
CV	EWS	16.53	116.61	- 21.15	present study
	activated carbon (apricot)	49.36	- 161.15	- 2.64	Onal, 2006
	charred saw dust	59.2	212.5	- 9.0	Chakraborty et al., 2005
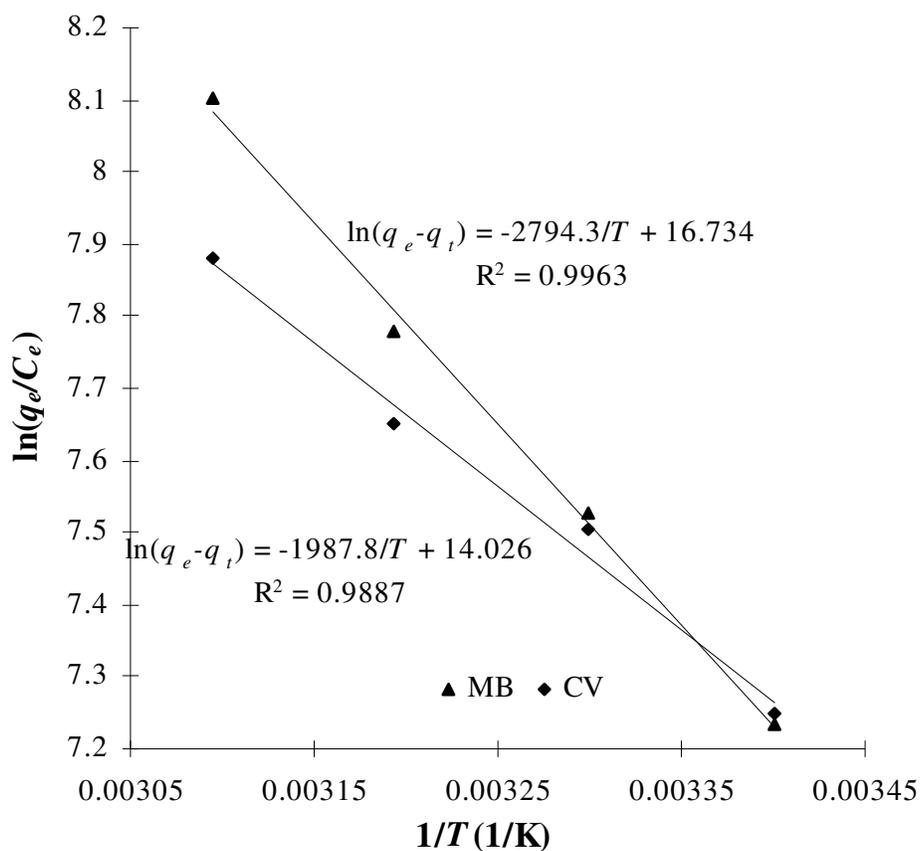
Figure 8. The plot of ln q_e/C_e vs. 1/T for the sorption of MB and CV by EWS (dye concentration: 400mg/dm^3; EWS dose: 3g/dm^3; contact time: 20 h; pH: natural).

from the following Gibbs-Helmholtz equation:

$$\Delta G° = \Delta H° - T\Delta S°$$

where $\Delta G°$ is the standard free energy (kJ/mol). The $\Delta G°$ values of MB and CV sorbed on EWS at 323.15 K as well as $\Delta H°$ and $\Delta S°$ values were presented in Table 3. The negative values of $\Delta G°$ and the positive values of $\Delta H°$ indicated that these sorption processes were spontaneous and endothermic. Because the process of dye diffusing into sorbent surface is energy-consuming, most

of dye sorptions are generally endothermic in nature.

SEM photographs of EWS

The SEM photographs of EWS before and after dye sorption were shown in Figure 9. The SEM photograph before dye sorption revealed the rough surface texture of EWS. After dye sorption, EWS presented the smooth surface texture because the dye molecules were trapped and sorbed on its surface.
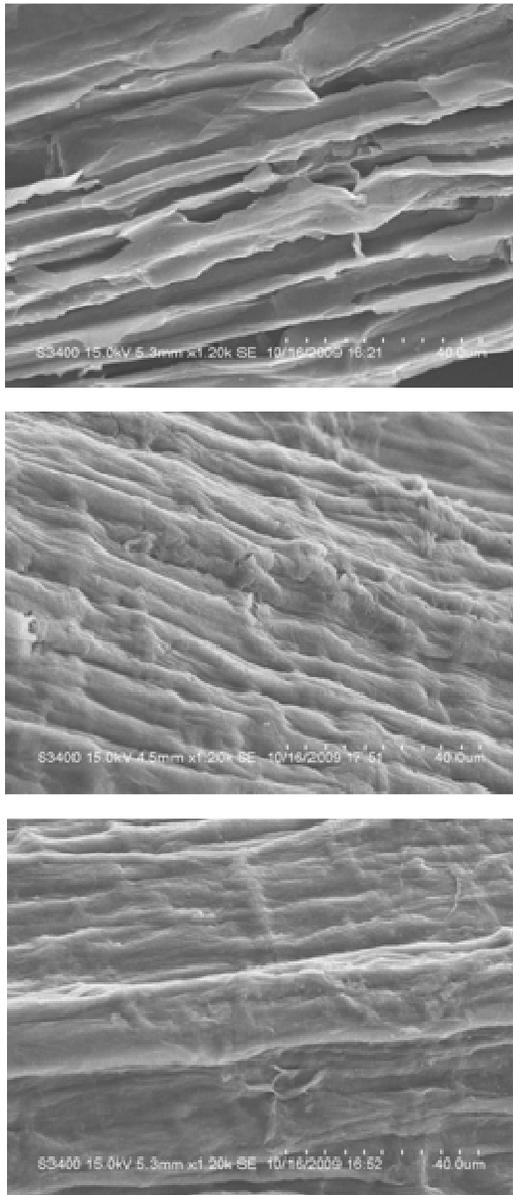
Figure 9. The SEM photographs of EWS (above: EWS; middle: after MB sorption; below: after CV sorption)

Conclusion

This study confirmed that EWS was an excellent basic dye sorbent. The isothermal data of MB and CV sorbed on EWS followed the Langmuir model better than the Freundlich model. The maximum sorption capacity (Q_m) of EWS for MB and CV was 129.87 and 112.36 mg/g, respectively. The dye sorption processes might be described by the pseudo-second-order kinetic model. The kinetic result of dye sorptions indicated that intraparticle diffusion was not the only rate control step. The thermodynamic study suggested that the dye sorptions were spontaneous and endothermic. The ion exchange

sorption maybe was the mechanism of dye sorbed on EWS.

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