Int. J. Environ. Res., 4(4):817-824, Autumn 2010
ISSN: 1735-6865

Equilibrium and Kinetic Studies on Sorption of Malachite Green using *Hydrilla Verticillata* Biomass

Rajesh Kannan, R.[1*], Rajasimman, M.[1], Rajamohan, N.[2] and Sivaprakash, B.[1]

[1] Environmental Engineering Laboratory, Department of Chemical Engineering, Annamalai University, Annamalai Nagar-608 002, Tamil Nadu, India

[2] Department of Chemical Engineering, Sohar University, Sohar, Sultanate of Oman

Received 3 April 2009; Revised 15 May 2010; Accepted 26 May 2010

ABSTRACT: In the present study, *Hydrilla verticillata* biomass was investigated as a novel biosorbent for the uptake of basic dye malachite green from its aqueous solution. Kinetic and equilibrium studies were carried out in batch process. Batch adsorption experiments were conducted to study the effect of pH, temperature, sorbent dosage, initial dye concentration, and contact time for the removal malachite green dye. The dye uptake was maximum for the initial pH of 8, temperature of 30°C, sorbent dosage of 0.55g, initial dye concentration 200mg/l and contact time – 150 min. The kinetic studies were well modeled using pseudo first order and second order with isotherm studies.

Key words: Malachite Green, *Hydrilla verticillata*, plant biomass, Equilibrium studies

INTRODUCTION

Synthetic dyes are extensively used in many industries such as the textile, leather, paper production, food technology, hair colorings, etc. Wastewaters discharged from these industries are usually polluted by dyes. Malachite green (MG) is most com-monly used for the dyeing of cotton, silk, paper, leather and also in manufacturing of paints and printing inks. Malachite green is widely used in distilleries for coloring purposes (Khattri *et al.,* 1999). In the recent decade ample attention has been paid to equilibrium and kinetic studies of various sorbents (Igwe *et al.,* 2008; Gharbani *et al.,* 2008; Goyal *et al.,* 2008; Shah *et al.,* 2009; Zvinowanda *et al.,* 2009). Mala-chite green has properties that make it difficult to remove from aqueous solutions and also toxic to major microorganisms (Papinutti *et al.,* 2006). Though the use of this dye has been banned in several countries and not approved by US Food and Drug Administration (Chang *et al.,* 2001), it is still being used in many parts of the world due to its low-cost, ready availability and efficacy and to lack of a proper alternative(Schnick *et al.,* 1988). Its use in the aquaculture practice in many countries, including India has not been regulated (Rahman *et al.,* 2005). Malachite green when discharged into receiving streams will affect the aquatic life and causes detrimental effects in liver, gill, kidney, intes-tine, gonads and pituitary gonadotrophic cells (Srivastava *et al.,* 2004). Therefore, the treatment of effluent containing such dye is of interest due to its esthetic impacts on receiving waters.

Various techniques have been employed for the removal of dyes from wastewaters. These methods include adsorp-tion, nano-filtrtion, electro kinetic coagulation, coagulation and precipitation, advanced chemical oxidation, electrochemical oxidation, ozonation, supported liquid membrane, liquid-liquid extraction and biological process (Mahmoud *et al.,* 2007). The adsorption process is one of the efficient methods to remove dyes from effluent due to its low initial cost, simplicity of design, ease of operation and insensitivity to toxic substances (Robinson *et al.,* 2001). Activated carbon is the most widely used adsorbent with great success due to its large surface area, micro-porous struc-ture, high adsorption capacity, etc. However, its use is limited because of its high cost. This has led to search for cheaper substi-tutes (Allen *et al.,* 2003). Various cheap adsorbents like wood and lignite have been used for the removal of colour and metal ions in effluents. Other materials include fly ash, rice husk, tree bark and human hair have been tested and reported to give encouraging results in several areas of application (Malik *et al.,* 2003). However the adsorption capacities of the above adsorbents are not very high. In order to improve the

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*Corresponding author E-mail:kannan_vrr007@yahoo.com efficiency of the adsorption processes, it is necessary to develop cheap and easily available adsorbents with high adsorption capacities. In a world of rapid assimilation of natural resources, any attempt at the utilization of agricultural waste augments the raw material stock and also provides additional employments and income to marginal farmers and landless agricultural labourers, especially in developing countries like India. It should be pointed out that various researchers from different parts of world have tried to use very divers materials as a means of adsorbent (Labidi, 2008; Lori *et al.,* 2008; Murugesan *et al.,* 2008, Nabi Bidhendi *et al.,* 2007; Rahmani *et al.,* 2009; Sahmoune *et al.,* 2009; Salim and Munekage, 2009, Singanan *et al.,* 2008).

Hydrilla verticillata, a submerged aquatic plant found widely in India, is listed as one of the most productive plants on earth and is considered as one of the world's worst aquatic plants. It forms dense mats that interfere with navigation, recreation, irrigation, and power generation. These mats competitively exclude native submerged and floating-leaved plants. Due to vegetative reproduction and extremely high growth rate, Hydrilla verticillata has attracted the attention of scientists to use it as a potential biomass for the removal of cadmium from its aqueous solution (Rahman *et al.,* 2005), because of its high growth yield and availability in large amount throughout the year and around the world. In the light of afore-mentioned studies, as Hydrilla is widely available, it could represent a cheap source of biosorbent for basic dyes (Low *et al.,* 1994). This research study is conducted to utilize the Hydrilla verticillata as a potential adsorbent to remove Malachite Green from its aqueous solutions.

MATERIAL & METHODS

The *Hydrilla verticillata* biomass used in this study was obtained from a pond near by Department of Chemical engineering, Annamalai University, Annamalainagar, Tamilnadu, India. The collected biomaterial was extensively washed with tap water to remove soil and dust and sliced into pieces. The sliced material was dried by exposure to the sunlight for 3 days and subsequently at 80ºC for 3 h in a hot air convection oven. The dried material was milled into a powder and was allowed to pass through a -65/+80 mesh opening size sieve. For further studies the sieved powder was treated with 2.0 N HCl for 24 h. After that, the samples were filtered and rinsed with distilled water. Treated material was dried again at 80ºC for 6 h, sealed in plastic bags, and stored in desiccators for use.

Malachite green was obtained from SD Fine Chemicals Ltd. (India) and was further used without any purification. All other reagents were of analytical reagent grade and were obtained from Qualigens Fine Chemicals, Mumbai, India. A calculated amount of the dye was dissolved separately in 1 L of deionized water to prepare stock solutions, which were kept in dark colored glass bottles. For batch study, an aqueous solution of this dye was prepared from stock solutions in deionized water. NaOH and HCl solutions were used as buffers for pH studies. The chemical structure of malachite green oxalate is shown in Fig.1 and the detailed information of the malachite green is given in Table 1.

The dye concentration in raw and treated sample was determined by UV-Vis (Elico, SL 164, Hydrabad, India) Spectrophotometer. The analyses were carried out at wavelength of 619 nm in a UV-Vis Spectrophotometer. A calibration plot for malachite green was drawn between percentage absorbance and standard dye solutions of various strengths. Runs were in triplicate. From the noted absorbance value the initial concentration, the concentration of the treated dye sample was determined. The effect of pH, quantity of biomass, initial concentration and temperature were monitored. The amount of equilibrium adsorption, q_e (mg/g), was calculated by:

$$q_e = \frac{V(C_o - C_e)}{M} \tag{1}$$

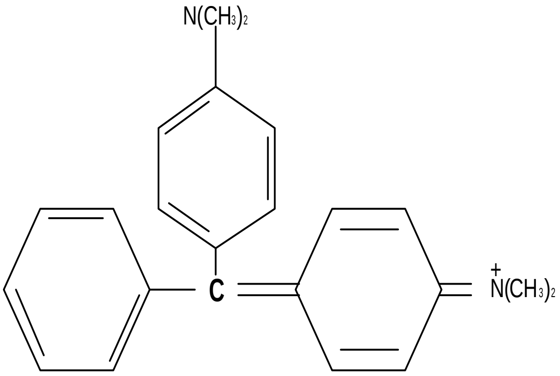

Fig. 1. Chemical structure of malachite green

Table 1. Information of the dye used

Name of dye	**Malachite Green**
CI name	Basic Green4 (BG4)
Color index number	42000
Empirical formula	$C_{23}H_{25}N_2ClC$
Molecular weight	365
Dye content	90%
λ max	619 nm

where C_0 and C_e (mg/L) are the liquid-phase concentrations of dye at initial and equilibrium, respectively. V(L) is the volume of the solution and M (g) is the mass of dry sorbent used.

RESULTS & DISCUSSION

In this work, the effect of pH on the MG adsorption onto Hydrilla verticillata Biomass was studied while the initial dye concentration, shaking time, amount of Hydrilla verticillata biomass and temperature were fixed at 100 mg/L, 200 min, 0.30 g and 30 °C, respectively. Solution pH would affect both aqueous chemistry and surface binding-sites of the adsorbent. The effect of pH on the adsorption of MG by the Hydrilla verticillata biomass is presented in Fig. 2. The equilibrium sorption capacity was minimum at pH 2 (11 mg/g) and increases monotonically up to pH 5, Further increase in pH leads to appreciable increase in % adsorption. The absence of sorption at low pH can be explained by the fact that at this acidic pH, H^+ may compete with dye ions for the adsorp-tion sites of adsorbent, thereby inhibiting the adsorption of dye. At higher solution pH, the Hydrilla verticillata biomass may get negatively charged, which enhances the adsorption of positively charged dye cations through electrostatic forces of attraction. Also a change of solution pH affects the adsorptive process through dissociation of functional groups on the adsorbent surface. Such behavior leads to a shift in equilibrium characteristics of adsorption process. A similar result of pH effect was also reported for the adsorption of Malachite green onto rattan saw dust (Hameed *et al.,* 2008).

The influence of temperature on the sorption of malachite green by the Hydrilla verticillata biomass was studied with a constant initial concentration of 100 mg/L and with a temperature range of 20°C to 50°C. The Effect of temperature on adsorption capacity is depicted in Fig. 3. The temperature profile indicates that as the temperature increases the sorption capacity increases to a maximum value and then decreases. This is because at very high temperature the biosorbent looses its property due to denaturation. The maximum sorption capacity was attained at 30°C. Similar result of Temperature effect was also reported for the adsorption of Basic dye onto chitosan (Chellababu et al., 2008).The effect of quantity of Hydrilla verticillata biomass dosage on the amount of color adsorbed was studied by agitating 200mL of 100mg/L dye solution with different amounts of sorbent addition such as 0.25, 0.35, 0.45 and 0.55g. All these studies were conducted at room temperature and at a constant speed of 200 rpm. Fig. 4 shows the effect of adsorbent dosage on the amount of dye adsorbed q_e (mg/g). It was observed that the amount of dye adsorbed varied with initial adsorbent dosage. The amount adsorbed decreased from 41.5 to 28.46 mg/g for an increase in sorbent dosage from 0.25 to 0.55g. An increase in % colour removal was observed with an increase in adsorbent dosage. The decrease in may be due to the solute transfer rate on to the adsorbent surface,i.e.,The amount of solute adsorbed onto unit weight of adsorbent get split with increasing adsorbent dosage.

The contact time between the dye molecules and the sor-bent is of significant importance in the dye treatment by sorption. The effect of contact time on the sorption of malachite green was studied for an initial dye concentration of 100 mg/l, a sorbent mass of 0.25 g, a solution volume of 100 mL, an agi-tation speed of 200 rpm, and a temperature of 30 °C. The effect of contact time on the removal of dye by the studied sorbent is shown in Fig. 5. The obtained results reveal that the uptake

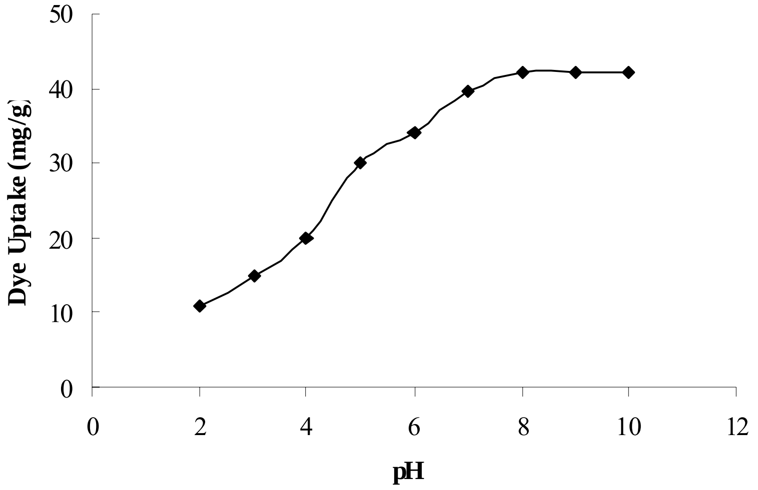

Fig. 2. Effect of pH on equilibrium uptake of MG (M = 0.30 g; V = 0.2 L; C_o = 100 mg/L)

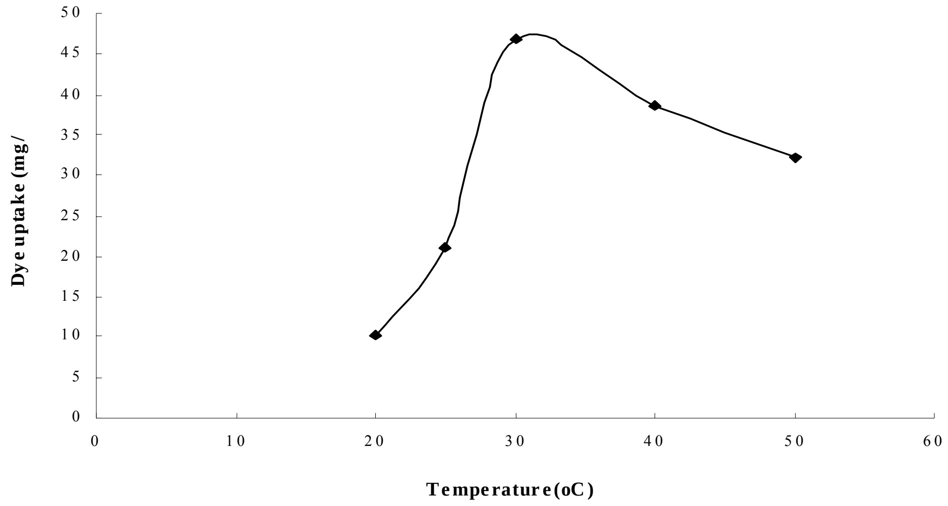

Fig. 3. Effect of temperature on equilibrium uptake of MG (M = 0.30g; V = 0.2 L; C_o= 100 mg/L)

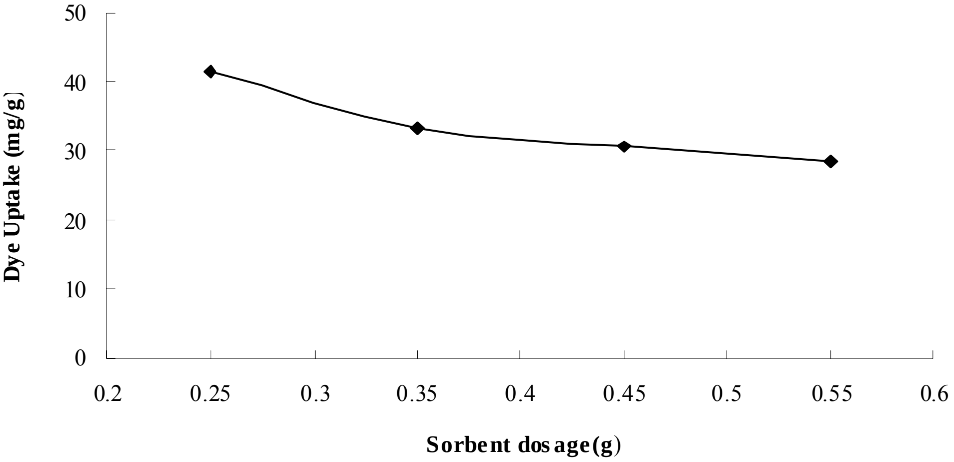

Fig. 4. Effect of sorbent dosage on equilibrium uptake of MG (V = 0.2; C_o = 100 mg/L)

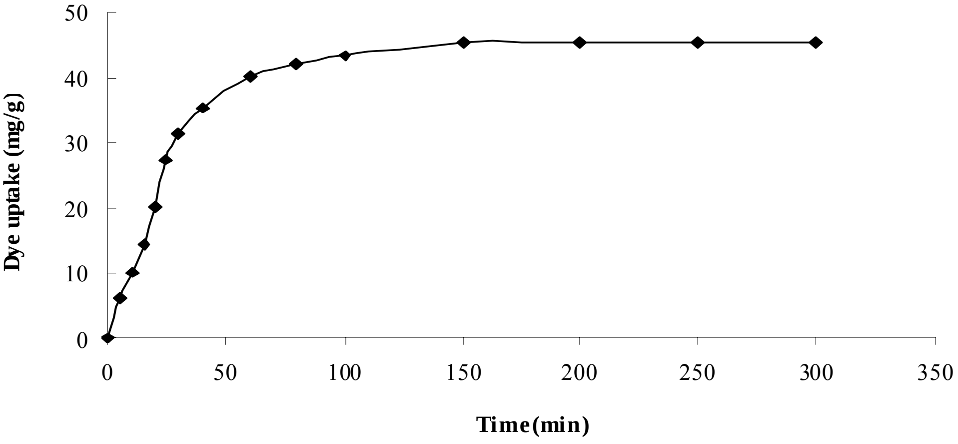

Fig. 5. Effect of contact time on sorption of MG dyes by the Hydrilla Verticillata biomass - initial dye concentration: 100 mg/L, sorbent mass: 0.55 g; agitation speed: 200 rpm)

of sorbate species is fast at the initial stage of the contact period (90 min), and thereafter, it becomes slower near the equilibrium. In between these two stages of the uptake, the rate of sorption is found to be nearly constant. This is obvious from the fact that a large number of vacant surface sites are available for sorption during the initial stage, and after a lapse of time, the remaining vacant surface sites are difficult to be occupied due to repul-sive forces between the solute molecules on the solid and bulk phases. The contact time necessary to reach equilibrium is about 150 min. At this point, the sorbed amount of dye by Hydrilla verticillata biomass is in a state of dynamic equilibrium with the amount of the dye desorbing from the sorbent. Additionally, the curve of contact time is single, smooth, and continuous leading to equi-librium. This curve indicates the possible monolayer coverage of dye on the surface of Hydrilla verticillata biomass, similar result of contact time effect was also reported for the adsorption of Malachite Green onto plane tree leaves (Oualid Hamdaoui et al., 2008).

Batch experiments were carried out by agitating with 100mL of dye solutions whose concentrations were 25, 50, 100,150 and 200mg/l at an optimum pH of 8.0 with 0.55g of Hydrilla verticillata biomass at room temperature. The speed of agitation was maintained constant at 200 rpm. The colour reduction profiles were obtained using the absorbance measurements.

Two commonly used isotherms, Langmuir (Langmuir, 1916) and Freundlich (Freundlich, 1906), were employed in the present study. The nonlinear Langmuir and Freundlich isotherms are represented by Eqs. (2) and (3):

$$q_e = K_F Ce^{1/n} \tag{2}$$

$$q_e = \frac{q_{\max} k_a C_e}{1 + k_a C_e} \tag{3}$$

where C_e (mg/L) is the equilibrium concentration, q_e (mg/g) is the amount of dye adsorbed at equilibrium, and q_m (mg/g) and K_a (L/mg) are Langmuir constants related to adsorption capacity and energy of adsorption, respectively. K_F (mg/g) (L/mg)$^{1/n}$ is the Fre-undlich adsorption constant and 1/n is a measure of the adsorption intensity.

Fig. 6 shows the fitted equilibrium data in Freundlich and Langmuir isotherms. The fitting results, i.e. isotherm parameters and the coefficients of determination, R^2, are shown in Table 2. It can be seen in Fig. 6 that Langmuir isotherm fits the data better than Fre-undlich isotherm. This is also confirmed by the high value of R^2 in case of Langmuir (0.9963) compared to Freundlich (0.961) and this indicates that the adsorption of MG on Hydrilla verticillata biomass takes place as monolayer

Table 2. Isotherm constants for MG on adsorption on Hydrilla verticillata biomass at 30°C

Langmuir isotherm	
Q_{max} (mg/g)	69.88
K_f (L/mg)	0.0201
R^2	0.9963
Freundlich isotherm	
K_a	3.641
n	1.736
R^2	0.961

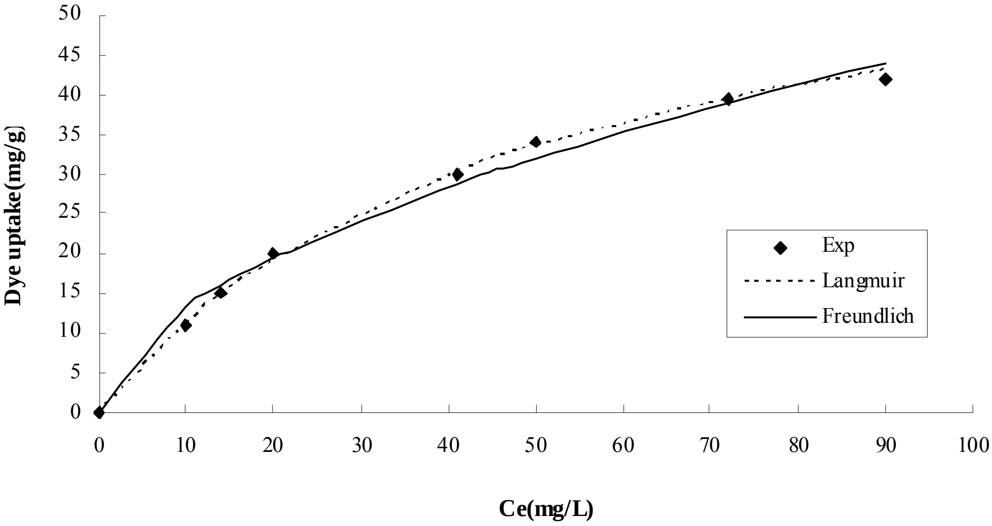

Fig. 6. Isotherm plot for MG adsorption on Hydrilla Verticillata biomass at 30°C

adsorption on a surface that is homogenous in adsorption affinity. The Hydrilla verticillata biomass adsorbent used in this work had a relatively large adsorption capacity (69.88 mg/g) compared to some other adsor-bents reported in the literature, Table 3 compares the adsorption capacity of different types of adsorbents used for removal of MG (Zhang *et al.,* 2008; Iqubal *et al.,* 2007; Tahir *et al.,* 2006; Mall *et al.,* 2005; Hema *et al.,* 2008). The most important parameter to compare is the Langmuir q_m value since it is a measure of adsorption capacity of the adsorbent. The value of q_m in this study is larger than those in most of previous works. This suggests that MG could be easily adsorbed on Hydrilla Verticillata biomass.

In order to investigate the adsorption processes of MG on Hydrilla verticillata, Lagergren's pseudo-first-order model (Eq. (4)) (Lagergren. 1898), and Ho's pseudo-second-order model (Eq. (5)) (Ho.1995) were used

$$q = q_e\left(1 - e^{-k_1 t}\right) \qquad (4)$$

$$q = \frac{q_e^2 k_2 t}{1 + q_e k_2 t} \qquad (5)$$

Where q_e (mg/g) is the amount of adsorbate adsorbed at equilib-rium, q (mg/g) is the amount of adsorbate adsorbed at time t, k_1 (L/min) is the rate constant of pseudo-first-order adsorption, k_2 (g/mg. min) is the rate constant of pseudo-second-order adsorption. The fittings of the experimental kinetic results to Eqs. (4) and (5) were done by nonlinear regression. The fitting results are shown in Figs. 7 and 8, and the values of the estimated parameters are presented in Table 4. The figures show that the adsorption rate (dq/dt) decreases with time until it gradually approaches the equi-librium state due to the continuous decrease in the driving force (q_e - q_t). The plots in Figs. 7 and 8 also demonstrate that the adsor-bate uptake q increases with increasing the initial concentration. It can be seen in Table 4 that the values of the coefficients of determi-nation, R^2 of the pseudo-first-order model are all higher than those of the second-order model, and also the estimated q_e values from the first-order model are much more accurate. The goodness of fit and the accurate prediction of q_e both indicate that the pseudo-first-order model better describes the adsorption of MG on Hydrilla Verticillata Biomass

Table 3. Comparison of adsorption capacities of various adsorbents for malachite green

Adsorbent	q_m (mg/g)	T (? C)	References
Hydrila Verticilata Biomass	69.88	30	This work
Arundo donax root carbon	8.70	30	(Zhang et al., 2008)
Activated charcoal	0.179	30	(Iqubal et al., 2007)
Bentonite clay	7.72	35	(Tahir et al., 2006)
Activated carbons			
Activated carbon commercial grade (ACC)	8.27	30±1	(Mall et al., 2005)
Laboratory grade activated Carbon(ACL)	42.18	30±1	(Mall et al., 2005)
Acid activated loe cost carbon	9.7377	30	(Hema et al., 2008)

Table 4. Kinetic models parameters for the adsorption of MG on Hydrilla Verticillata biomass at 30°C and different initial MG concentrations (C_0: mg/L; q_e: mg/g; k_1: L/min; k_2: g/mg min)

C_0 (mg/L)	pseudo first order			pseudo-second-order		
	q_{exp}	q_e	R^2	q_e	k_2 x 10^3	R^2
25	11.0	11.06	0.9963	12.2	9.149	0.9537
50	20.00	20.0	0.965	22.62	3.67	0.972
100	30.05	30.10	0.970	33.22	2.83	0.966
150	40.1	40.2	0.9839	44.44	1.99	0.983
200	47.08	47.1	0.9833	50.51	2.74	0.988

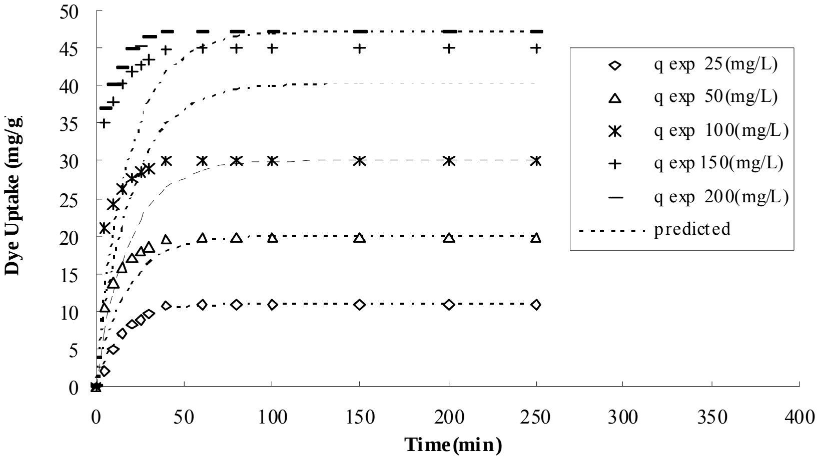

Fig. 7. Fitting with pseudo-first order model for MG on Hydrilla verticillata biomass at different initial concentrations

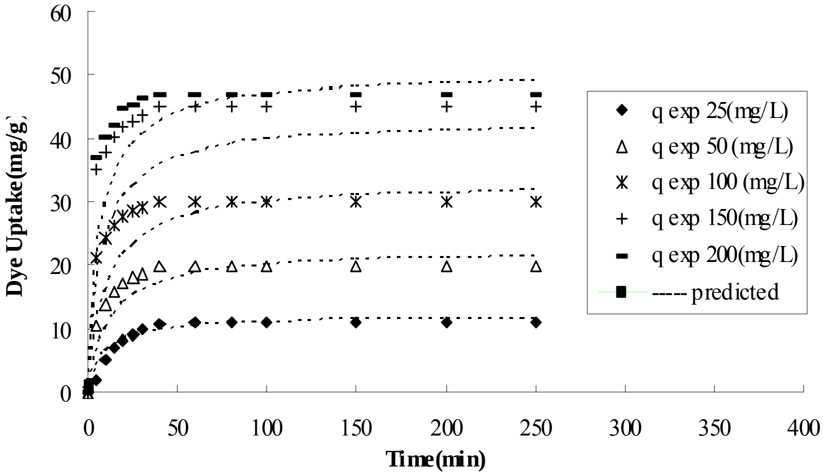

Fig. 8. Fitting with pseudo-second order model for MG on Hydrilla Verticillata biomass at different initial concentrations at 30°C

CONCLUSION

The results obtained in this study indicate the Hydrilla verticillata biomass can be successfully used for the removal of hazardous dye, malachite green from aqueous solutions. The batch sorption process is found depend upon pH, temperature, sorbent dosage and initial dye concentration. The Langmuir adsorption isotherm was found to have the best fit to the experimental data, suggesting monolayer adsorption on a homogeneous surface. The kinetic data shows that pseudo-first order model is obeyed better than pseudo-second order model since second order model provide high degree of correlation with the experimental data at various experimental condition. Thus, it can be concluded that the waste biomaterial Hydrilla verticillata Biomass can be used as excellent sorbent for the removal of dyes from waste water.

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