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Nitric acid-treated bamboo waste as low-cost adsorbent for removal of cationic dye from aqueous solution

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ABSTRACT

The feasibility of nitric acid-treated bamboo waste (NBW) for the removal of methylene blue (MB) from aqueous solutions was investigated. Batch adsorption studies were conducted to study the effects of contact time (0–180 min), initial concentration (45–375 mg/L) and solution pH (2–10) on the removal of dye at 30°C. The experimental data were analyzed by using four different types of linearized Langmuir and the Freundlich isotherms. Equilibrium data fitted well with the type 1 Langmuir model, yielding maximum monolayer MB adsorption capacity of 87.719 mg/g. The kinetic data were found to conform to pseudo-second-order kinetic model with good correlation. Intraparticle diffusion model was further tested to identify the diffusion mechanism. The scanning electron micrographs showed that MB was adsorbed on the rough surface of NBW. The NBW prepared in this study was shown to be a promising low-cost material for adsorption of MB from aqueous solutions.

Keywords: Adsorption; Bamboo waste; Isotherm; Kinetics; Methylene blue

1. Introduction

Textile industry is growing in Malaysia and consequently produces large amounts of color effluent that is unacceptable under Malaysian environmental regulation and should be treated before discharge. The treatment of textile effluents is of interest due to their toxic and esthetic impacts on receiving waters. There are many treatment processes applied for the removal of dyes from wastewater. A critical review on current treatment technologies with a proposed alternative was reported by Robinson et al. [1].

Adsorption process using commercial activated carbons is very effective for removal of dyes from wastewater, but its high cost has motivated the search for alternative materials. Most of the adsorption studies have been focused on untreated plant/agricultural wastes such as orange peel [2], rice husk [3], oil palm trunk fiber [4], Aspergillus fumigatus [5], wheat straw [6], durian (Durio zibethinus Murray) peel [7], Eucalyptus bark powder [8], almond shells [9], clay materials [10], pomelo (Citrus grandis) peel [11], Luffa cylindrica fibers [12], Parthenium biomass [13], castor seed shell [14] and granular kohlrabi peel [15]. Recently, an extensive list of adsorbent literature for dye removal has been compiled by Allen and Koumanova [16].

Although the application of untreated agricultural waste attracted much research recently, two problems are associated with it: low adsorption capacity and release of soluble organic compounds contained in the

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Table 1
Summary of modified materials as adsorbents for the removal of dyes from aqueous solutions

Adsorbent	Modifying agent	Dye	Maximum adsorption capacity (mg/g)	References
Bamboo waste	HNO_3	MB	87.719	This work
Beech sawdust	None	MB	9.78 (7.93–12.8)	[17]
Beech sawdust	20% w/v $CaCl_2$ at 23°C	MB	13.02 (9.89–18.7)	[17]
Beech sawdust	20% w/v $CaCl_2$ at 100°C	MB	16.05 (10.9–30.5)	[17]
Coconut coir pith	Hexadecyltrimethylammonium (HDTMA) bromide	Direct Red 12B	76.30	[18]
Parthenium hysterophorus	Sulphuric acid	MB	39.68	[19]
Parthenium biomass	Formaldehyde	Rhodamine-B	28.82	[20]
Parthenium carbon	Phosphoric acid	Rhodamine-B	59.17	[20]
White-rot fungus biomass	None	Reactive Red-120	117.84	[21]
White-rot fungus biomass	Heat	Reactive Red-120	182.97	[21]
White-rot fungus biomass	HCl	Reactive Red-120	138.61	[21]
White-rot fungus biomass	NaOH	Reactive Red-120	57.22	[21]
Bentonite	Cetyltrimethylammonium bromide	Benzopurpurin 4B (Direct Red 2)	109.89	[22]
Diatomite	H_2SO_4	MB	126.60	[23]
Kaolinite clay	None	Aniline blue	1.67	[24]
Kaolinite clay	Sodium Tetraborate	Aniline blue	2.00	[24]

plant materials. Hence, the objective of the treatment process is to improve the surface characteristics of the adsorbent. Therefore, such materials should be modified or treated before being applied for the removal of dyes from aqueous solutions. In general, chemically treated plant wastes exhibit higher adsorption capacities than the untreated materials. The types of chemicals used for different adsorbents reported in the literature, together with their maximum adsorption capacities are listed in Table 1.

In this work, an attempt to chemically treat bamboo waste, an abundant and inexpensive natural resource in Malaysia, as a potential low-cost adsorbent for MB removal has been made. Bamboo is the fastest growing renewable woody plant on this planet. It grows one third faster than the fastest growing tree. Some species can grow up to one meter per day [25]. This growth pattern makes it easily accessible in a minimal amount of time. The size of bamboo ranges from miniatures to towering culms of 60 m. Bamboo has been used as the structural material for steps at construction sites in China, India, Malaysia and other countries because it is a strong, tough and low-cost material. Bamboo is currently used to make thousands of products such as indoor and out door furniture, mat, flooring accessories, fences, curtains, paneling, chopsticks and toothpicks etc. Therefore, a huge amount of bamboo waste is expected to be generated. Conversion of such waste to a value-added product such as adsorbent for dye removal will be rather useful.

Therefore, the objective of this study was to evaluate the potentiality of using nitric acid-treated bamboo waste for the adsorption MB from aqueous solutions under different experimental conditions.

2. Materials and methods

2.1. Adsorbate

The MB (C.I. basic blue 9) used in this work was purchased from Sigma-Aldrich (M) Sdn Bhd, Malaysia. MB was chosen in this study because of its known strong adsorption onto solids. MB has a molecular weight of 373.90 g/mol. The maximum adsorption wavelength of this dye is 668 nm.

2.2. Preparation of adsorbent

Bamboo waste (pieces of 3–12 cm length) used in this work was obtained from local furniture shop, Penang, Malaysia. The pieces were washed, dried and ground to desired mesh size (1–2 mm). A portion of the sized sample (20 g) was refluxed with 0.5M nitric acid (HNO_3) for 20 min. It was then washed repeatedly with hot distilled water until pH of the washing solution was in near neutral range (pH 6.8–7.0). Thereafter, the NBW prepared was dried at 60°C for 24 h in an oven. The dried sample was stored in an airtight container for further use.

Scanning electron microscopy (SEM) analysis was carried out on the NBW prepared to study its surface texture before and after MB adsorption.

Table 2
Isotherms and their linearized forms

Isotherm		Linear form	Plot	References
Type 1 Langmuir	$q_e = \frac{q_m K_a C_e}{1 + K_a C_e}$	$C_e/q_e = C_e/q_m + 1/K_a q_m$	C_e/q_e vs. C_e	[26]
Type 2 Langmuir		$1/q_e = (1/K_a q_m C_e) + 1/q_m$	$1/q_e$ vs. $1/C_e$	
Type 3 Langmuir		$q_e = q_m - q_e/K_a C_e$	q_e vs. q_e/C_e	
Type 4 Langmuir		$q_e/C_e = K_a q_m - K_a q_e$	q_e/C_e vs. q_e	
Freundlich	$q_e = K_F C_e^{1/n}$	$\ln q_e = \ln K_F + 1/n \ln C_e$	ln q_e vs. ln C_e	[27]

2.3. Batch equilibrium studies

Batch adsorption studies were carried out by adding a fixed amount of NBW (0.40 g) into a number of 250 mL-stoppered glass Erlenmeyer flasks containing a definite volume (200 mL in each flask) of different initial concentrations (45–375 mg/L) of dye solution without changing solution pH and at constant temperature of 30°C. The flasks were placed in a thermostated water-bath shaker and agitation was provided at 130 rpm for 180 min. At time $t = 0$ and equilibrium, the dye concentrations were measured using a double beam UV-visible spectrophotometer (Shimadzu, Model UV 1601, Japan) at 668 nm wavelength. Prior to the measurement, a calibration curve was obtained by using the standard MB solution with known concentrations.

The amount of adsorption at equilibrium, q_e (mg/g), was calculated by:

$$q_e = (C_0 - C_e)V/W, \tag{1}$$

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

The dye removal (%) can be calculated as follows:

$$\text{Removal percentage} = ((C_0 - C_e)/C_0) \times 100. \tag{2}$$

2.4. Effect of solution pH

The effect of the pH of the dye solution on the amount of dye adsorbed was investigated in the pH range of 2–10 at 30°C. The solution pH was adjusted with 0.1 N NaOH and 0.1 N HCl solutions. The pH was measured by using a pH meter (Ecoscan, EUTECH Instruments, Singapore). 200 mL of a fixed initial concentration of dye solution (60 mg/L) at different pH values was agitated with 0.40 g of NBW in a water-bath shaker. Agitation was provided at a constant speed of 130 rpm for 180 min.

2.5. Batch kinetic studies

The procedures of batch kinetic experiments were basically identical to those of equilibrium tests. The aqueous samples were taken at preset time intervals, and the concentrations of dye were similarly measured. All the kinetic experiments were carried out at 30°C. The amount of sorption at time t, q_t (mg/g), was calculated by:

$$q_t = (C_0 - C_t)V/W, \tag{3}$$

where C_t (mg/L) is the liquid-phase concentrations of dye at any time, t.

3. Theory of adsorption isotherm and kinetics

3.1. Isotherm models

Adsorption isotherm is basically important to describe how solutes interact with adsorbents, and is critical in optimizing the use of adsorbents. Two adsorption isotherms, the Langmuir [26] and the Freundlich [27] were tested. The linearized forms of the two isotherms are shown in Table 2. The Langmuir constants q_m and K_a values can be calculated from the plot of C_e/q_e versus C_e, $1/q_e$ versus $1/C_e$, q_e versus q_e/C_e, and q_e/C_e versus q_e for type 1, type 2, type 3 and type 4 Langmuir isotherms, respectively. C_e is the equilibrium concentration (mg /L), q_e is the amount of dye adsorbed at equilibrium (mg/g) whereas q_m (mg/g) and K_a (L/mg) are Langmuir constants related to adsorption capacity and energy of adsorption, respectively.

Similarly the Freundlich isotherm constants, K_F and $1/n$ can be calculated from the plot of ln (q_e) versus ln

(C_e). K_F and n are Freundlich constants which are indicators of adsorption capacity and adsorption intensity, respectively.

3.2. Kinetics models

The most common models used to fit the kinetic sorption experiments are

Lagergren's pseudo-first order model (Eq. (4)) [28] and pseudo-second-order model

(Eq. (5)) [29] and these models were used to fit the kinetic data of the present work:

$$\log(q_e - q_t) = \log q_e - (k_1/2.303)t, \tag{4}$$

$$t/q_t = 1/k_2 q_e^2 + (1/q_e)t \tag{5}$$

where q_e (mg/g) and q_t (mg/g) are the amount of adsorbate adsorbed at equilibrium and at time t, respectively. k_1 (1/min) and k_2 (g/mg min) are the pseudo-first-order and pseudo-second-order adsorption rate constants, respectively.

3.3. Intraparticle diffusion model

In order to investigate the mechanism of the MB adsorption on NBW, intraparticle diffusion-based mechanism has been studied. The most commonly used technique for identifying the mechanism involved in the adsorption process is by fitting an intraparticle diffusion plot. It is an empirically found functional relationship, common to the most of adsorption processes, where uptake varies almost proportionally with $t^{1/2}$ rather than with the contact time t, according to the theory proposed by Weber and Morris [30]:

$$q_t = k_{pi} t^{1/2} + C_i, \tag{6}$$

where k_{pi} (mg/g min$^{1/2}$), the rate parameter of stage i, is obtained from the slope of the straight line of q_t versus $t^{1/2}$.

4. Results and discussion

4.1. Effect of solution pH on dye uptake

Fig. 1 shows the effect of solution pH on equilibrium adsorption uptake of MB on NBW at 60 mg/L initial MB concentration and at 30°C. It can be seen that the adsorption of MB was minimum at pH 2, slightly increased with pH up to pH 3 and then remained constant over the solution pH range of 4–10. The possible reason for the low adsorption uptake of MB on the NBW at pH < 3 was that the surface charge of NBW became positively charged, thus making H^+ ions compete effectively with dye cations, causing a decrease in the amount of dye adsorbed. Similar behavior was observed for MB adsorption on another agricultural waste, broad bean peels [31].

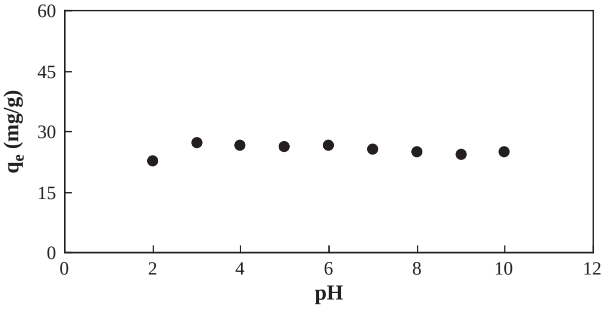

Fig. 1. Effect of pH on equilibrium uptake of MB (W= 0.40 g; V = 0.20 L; C_0 = 60 mg/L, T = 30°C).

4.2. Effect of contact time and initial dye concentration

The adsorption of MB on NBW was studied at different initial MB concentrations (45–375 mg/L). Fig. 2 shows the result for effect of initial concentration on adsorption of MB on NBW at 30°C. It was observed that the dye uptake was rapid for the first 20 min and thereafter proceeded at a slower rate and finally attained equilibrium. The equilibrium adsorption increased from 10.60 to 56.05 mg/g, with increasing initial MB concentration from 45 to 375 mg/L. However, as the initial MB concentration increased from 45 to 375 mg/L, the NBW removal efficiency of MB from solution was found to decrease from 93.33% to 86.00%.

The adsorption of MB at low initial concentrations (45–70 mg/L) attained adsorption equilibrium in less than 80 min, while at higher initial MB concentrations

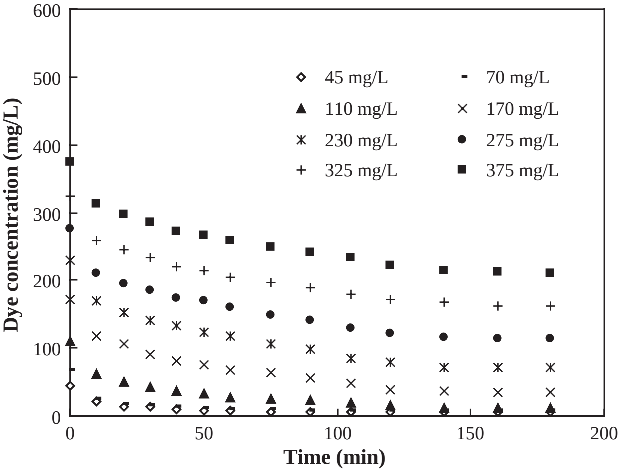

Fig. 2. Effect of initial concentration and contact time on MB adsorption (W = 0.40 g; V = 0.20 L; T = 30°C).

(110–375 mg/L), the time necessary to reach equilibrium was 150 min. However, the experimental data were measured at 180 min to ensure that full equilibrium was attained. From the literature, data on the adsorption kinetics of MB on various adsorbents have shown a similar range of adsorption rates. The study of Hameed and El-Khaiary [31] also showed that the adsorption of MB on broad bean peels reached equilibrium in less than 200 min. However, an adsorption equilibrium time of around 30 h was required for the adsorption of MB on activated carbon prepared from coconut husk [32].

4.3. Isotherm analysis

The equilibrium data for MB on NBW were modeled with four linearized expressions of the Langmuir and the Freundlich model. The details of the four types of linearized Langmuir isotherms and the method applied to estimate the Langmuir constants, q_m and K_a from these plots are given in Table 2 whereas the values obtained for the Langmuir constants are presented in Table 3. The highest value of the coefficient of correlation ($R^2 = 0.9983$) obtained from type 1 Langmuir expression indicated that type 1 Langmuir provided the best fit to the experimental data of MB on NBW among all the four linearized expressions studied.

The essential characteristics of the Langmuir isotherm can be expressed in terms of a dimensionless constant separation factor, R_L that is given by Eq. (7) [33]:

$$R_L = 1/(1 + K_a C_0) \tag{7}$$

where C_0 (mg/L) is the highest initial concentration of adsorbate and K_a (L/mg) is the Langmuir constant. The value of R_L indicates the shape of the isotherm to be either unfavorable ($R_L > 1$), linear ($R_L = 1$), favorable ($0 < R_L < 1$) or irreversible ($R_L = 0$). R_L value for the adsorption of MB on NBW was found to be 0.0305, indicating that the adsorption was a favorable process.

Table 3
Isotherm parameters for removal of MB on NBW at 30°C

Isotherm	Parameters	Values
Type 1 Langmuir	q_m (mg/g)	87.719
	K_a (L/mg)	0.0848
	R^2	0.9983
Type 2 Langmuir	q_m (mg/g)	102.041
	K_a (L/mg)	0.0521
	R^2	0.9684
Type 3 Langmuir	q_m (mg/g)	14.3840
	K_a (L/mg)	0.0695
	R^2	0.8911
Type 4 Langmuir	q_m (mg/g)	94.974
	K_a (L/mg)	0.0620
	R^2	0.8911
Freundlich	K_F	15.7462
	n	2.9011
	R^2	0.8494

The equilibrium data were further analyzed using the linearized form of Freundlich isotherm, by plotting ln q_e versus ln C_e. The calculated Freundlich isotherm constants and the corresponding coefficient of correlation are shown in Table 3. The coefficient of correlation ($R^2 = 0.8494$) was lower than that obtained from Langmuir model. The result showed that the value of n was greater than unity ($n = 2.9011$), indicating that MB was favourably adsorbed on NBW. The magnitude of Freundlich constant indicated easy uptake of MB from aqueous solution. From Table 3, it can be concluded the overall type 1 Langmuir was the most suitable model for the experimental data which indicated that the adsorption of MB on NBW took place as monolayer adsorption on a surface that was homogenous in adsorption affinity. The present result was in agreement with those of Hameed et al. [11] for the adsorption of MB on pomelo (*Citrus grandis*) peel in a batch system. The maximum MB adsorption capacity of NBW prepared in this work was found to be 87.719 mg/g. The comparison of adsorption capacity of NBW with other adsorbents found in the literature [17,19,23] for MB adsorption which are presented in Table 1 indicates that the NBW is very effective for MB adsorption.

4.4. Adsorption kinetics

The experimental kinetic data of adsorption studies were applied to the pseudo-first and pseudo-second order kinetic models. From the slopes and intercepts of the plots of log ($q_e - q_t$) versus t obtained at the initial concentrations of 45, 70, 110, 170, 230, 275, 325 and 375 mg/L at 30°C, first-order rate constant (k_1) and q_{eq} values were determined. The rate constants, k_1, evaluated from these plots with the correlation coefficients obtained are listed in Table 4. It can be seen that the experimental data deviated considerably from the theoretical data after this short period. This suggested that the adsorption system studied was not a pseudo-first-order process. Therefore, the experimental kinetic data were further analyzed using the pseudo-second-order model. By plotting t/q_t against t for different initial MB concentration as shown in Fig. 3, a straight line was obtained in all cases and by using Eq. (5), the second-order rate constant (k_2) and q_e were determined from the plots. The values

Table 4
Comparison of the pseudo-first-order and pseudo-second-order adsorption rate constants, and calculated and experimental q_e values for different initial MB concentrations

Initial concentration (mg/L)	$q_{e,exp}$ (mg/g)	Pseudo-first-order kinetic			Pseudo-second-order kinetic		
		k_1 (1/min)	$q_{e,cal}$ (mg/g)	R^2	k_2 (g/mg min)	$q_{e,cal}$ (mg/g)	R^2
45	19.950	0.007469	11.918	0.9903	0.006469	20.964	0.9997
70	30.120	0.005167	7.752	0.8918	0.006797	30.969	0.9999
110	48.415	0.004082	29.813	0.9862	0.00108	53.191	0.9982
170	68.693	0.007078	48.217	0.9895	0.000419	80.000	0.9933
230	79.110	0.006209	120.171	0.8907	0.000314	94.339	0.9898
275	80.910	0.004907	86.596	0.8981	0.000331	95.238	0.9897
325	81.500	0.003995	69.952	0.9674	0.000342	95.238	0.9915
375	82.025	0.004863	94.455	0.9056	0.000310	96.154	0.9824

of correlation coefficient were greater than 0.9824 and the theoretical, $q_{e,cal}$ values were closer to the experimental, $q_{e,exp}$ values at various initial MB concentrations (Table 4). Thus, it can be concluded that the pseudo-second-order kinetic model provided a better correlation for the adsorption of MB on NBW at different initial MB concentrations as compared to the pseudo-first-order model. These results suggested that the second-order mechanism was predominant and that chemisorption might be the rate-limiting step that controlled the adsorption process.

The pseudo-first-order and pseudo-second-order kinetic models could not identify the diffusion mechanism, therefore the kinetic results were then analyzed by using the intraparticle diffusion model. Weber and Morris plot [30] (q_t versus $t^{0.5}$) was used to investigate the intraparticle diffusion mechanism (Fig. 4). If the intraparticle diffusion was the only rate-controlling step, the plot passed through the origin; if not, the boundary layer diffusion controlled the adsorption to some degree [34]. As seen from Fig. 4, the plots were not linear over the whole concentration and time range, implying that more than one process affected the adsorption. Similar behaviour was reported for MB adsorption on palm kernel fibre [31].

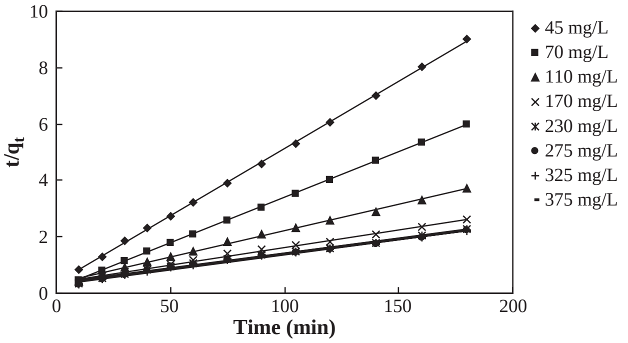

Fig. 3. Pseudo-second-order kinetic for adsorption of MB on NBW.

4.5. SEM analysis of NBW

Fig. 5 shows the SEM images (300×) for NBW prepared in this work before MB adsorption and after 180 min of adsorption process. It can be seen from Fig. 5(a) that NBW has a rough surface with a heterogeneous pores and cavities where there is a good possibility for MB dye molecules to be trapped and adsorbed on this surface. Fig. 5(b) in the other hand reveals that the surface morphology of the NBW after MB adsorption is different from the unused sample which is the indication of dye adsorption.

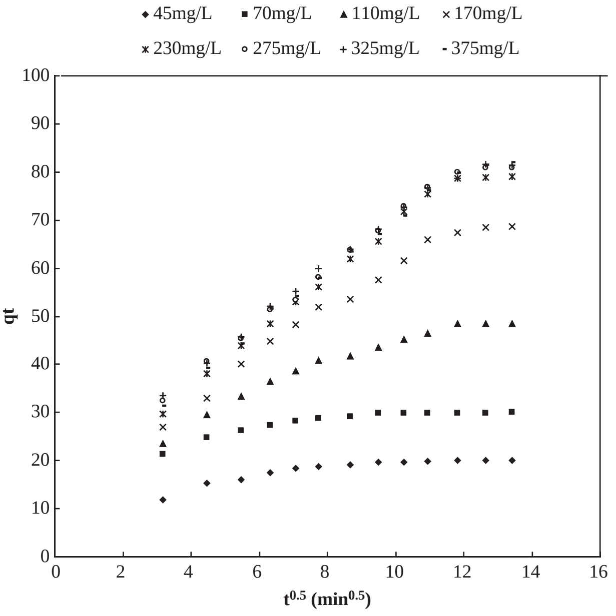

Fig. 4. Intraparticle diffusion plot for adsorption of MB on NBW for different initial MB concentrations.

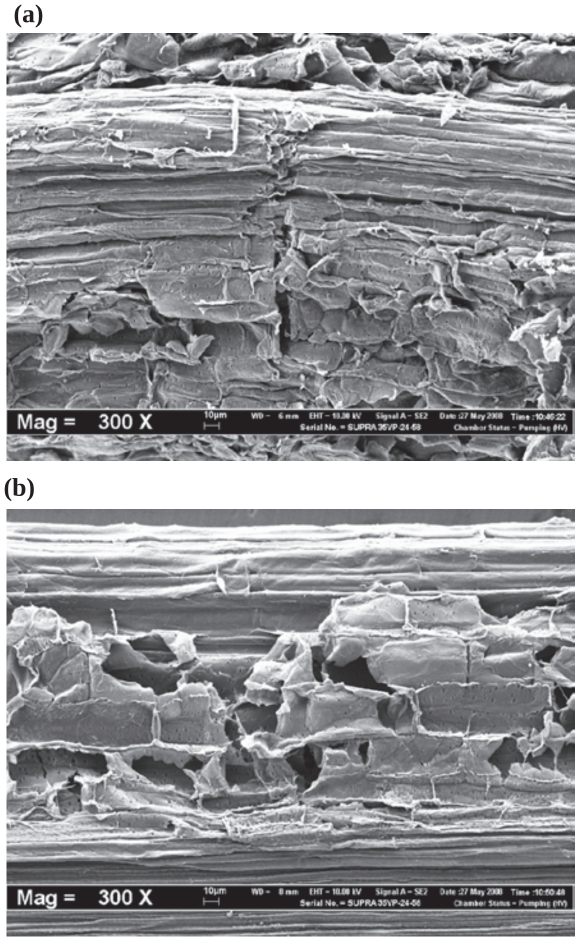

Fig. 5. SEM micrographs of NBW (magnification: 300x): (a) before dye adsorption and (b) after dye adsorption.

5. Conclusions

This investigation examined the adsorption performance of nitric acid-treated bamboo waste for MB over a wide range of concentrations. The equilibrium data were fitted to four different types of linearized Langmuir isotherm and the Freundlich isotherm models. The type 1 Langmuir adsorption isotherm was found to have the best fit to the experimental data, yielding a maximum adsorption capacity of 87.719 mg/g. In addition, the kinetic data were best described by the pseudo-second-order kinetic model. The above results confirmed the potentiality of NBW for application in removing MB from aqueous solutions.

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