Preparation and Characterization of Activated Carbon from *Lepironia articulata* (Retz.) Domin Waste as an Adsorbent for Methylene Blue Dye Removal from Wastewater

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In this study, the optimum conditions for using activated carbon from *Lepironia articulata* (Retz.) Domin waste as an adsorbent were determined to remove dye pollutants from wastewater. The characteristics of the obtained activated carbon were assessed by performing energy dispersive X-ray (EDX), scanning electron microscopy (SEM) and Fourier transform infrared spectroscopic (FTIR) techniques. The best optimum conditions for removing dye pollutants from wastewater were obtained at 30 mg/L methylene blue dye, 0.02 g of adsorbent and a contact time of 90 min. The maximum adsorption capacity of the activated carbon was 69.44 mg/g and the percentage removal achieved was more than 90%. The methylene blue dye adsorption data of the adsorbent were fitted to the Langmuir isotherm model. The prepared adsorbent was also applied for the removal of different colour dyes from real wastewater samples and found that the percentage removal was 71.3%, 62.7%, 66.3%, 68.4% and 72.0% for dark blue dye, red dye, pink dye, sky blue dye and yellow dye, respectively. These results indicated that the obtained activated carbon is highly effective and suitable adsorbent for removing different types of colour dyes and toxic organic substances from the wastewater.

Keywords: *Lepironia articulata* (Retz.) Domin, Activated carbon, Dye pollutant removal, Wastewater.

INTRODUCTION

As a result of global urbanization and industrialization, the quantity of toxins dumped into the water reservoirs has increased considerably [1]. Water is required for textile wet processing to remove impurities from fibers and for dyeing, printing and other finishing processes. The textile sector also uses various chemicals specifically, dyestuffs are a cause for concern. Dyes affect landfills and aquatic habitats as they are a source of heavy metals, salts, adsorbed organic halogens (AOX) and colour [2]. Therefore, methods for the effective removal of dyes need to be developed.

Many techniques have been developed that can be used for removing dry matter from wastewater, such as adsorption [3], photocatalytic degradation [4,5], sonocatalytic degradation [6,7], electrochemical methods [8], electrocoagulation [9], reverse osmosis [10], ultrafiltration mixed matrix membranes [11] and electro-Fenton processes [12]. Out of all of these appro-

aches, the adsorption is the one that is utilized rather frequently. It eliminates contaminants in an efficient manner regardless of the concentration of the pollutants [13]. Hence, several researchers have synthesized different types of adsorbent materials that are efficient and economical, such as chitosan cenosphere nanocomposite [14], graphene oxide [15], magnetic marble dust adsorbent [16] and activated carbon [17]. Specifically, activated carbon is commonly used as an adsorbent for wastewater treatment and is prepared from various types of materials. For example, activated carbon can be produced from mixed wastes of oil palm frond and palm kernel shell [18], mangosteen (*Garcinia mangostana*) peels [19], dry leaf waste of almond trees [20], stems of *Phyllanthus reticulatus* [21], dead lichen (*Pseudevernia furfuracea* (L.) Zopf.) [22] and black cumin seeds [23].

Lepironia articulata (Retz.) Domin waste is considered as agricultural waste that can be used as a raw material for preparing activated carbon due to its abundance as a source of

carbon. In other studies, various agricultural wastes were prepared and used as activated carbon, such as oil palm frond and palm kernel shell [18], black cumin seeds [23], rice husk [24], subabul (*Leucaena leucocephala*) sawdust waste [25] and bark of pine [26]. However, no study has investigated the use of *Lepironia articulata* (Retz.) Domin waste as a source of activated carbon. Therefore, in this study, we prepared and characterized activated carbon from *Lepironia articulata* (Retz.) Domin waste as an adsorbent for removing dyes from wastewater. Various techniques, such as scanning electron microscopy (SEM), energy dispersive X-ray spectroscopy (EDX), ultraviolet-visible spectroscopy (UV-Vis) and attenuated total reflectance-Fourier transform infrared (ATR-FTIR) spectroscopy were used to characterize the obtained activated carbon. Additionally, various parameters for removing dyes, including the concentration of dyes, the adsorbent dosage and contact time, were evaluated.

EXPERIMENTAL

Preparation of activated carbon adsorbent: *Lepironia articulata* (Retz.) Domin waste (LDW) was used as raw material for preparing activated carbon. All samples were collected from the Pak Phanang district of Nakhon Si Thammarat province, Thailand. The collected LDW was washed several times with water to remove dust and other impurities and dried in sunlight for a week, followed by vacuum-drying at 80 ℃ for 24 h. The dried LDW was crushed to form a powder, which was carbonized at 400 ℃ for 2 h under a continuous stream of argon gas. The carbonized product was soaked for 12 h in an aqueous solution of 10% (w/v) NaCl to activate it. The final sample was then heated at 850 ℃ for 2 h in the presence of argon gas. The activated carbon was washed several times with water to maintain neutral pH. The as-obtained sample was dried at 80 ℃ for 24 h. Finally, the formation of activated carbon was confirmed by using the SEM, EDX and FTIR techniques.

Batch adsorption studies: The effects of experimental parameters, such as the concentration of methylene blue (MB) dye, the dosage of adsorbent and the contact time required for the adsorptive removal of methylene blue dye were studied in a batch mode of operation. For batch adsorption of methylene blue dye, the adsorbents (0.01-0.12 g) were accurately weighed and placed in a 125 mL conical flask. Then, 50 mL of methylene blue dye solution (5-40 mg/L) was added and shaken using an orbital shaker at approximately 150 rpm at ambient temperature and different times of contact (0-180 min). After the solution was shaken for a specific period, the adsorbents were separated from the solutions by centrifugation for 5 min and the supernatants were analyzed using a spectrophotometer at a maximum wavelength (λ_{max}) of 665 nm. All the experiments were conducted in triplicate under the same conditions. The adsorption capacity (q_e, mg/g) of dye at an equilibrium state was determined using eqn. 1:

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

where C_o represents the initial concentration (mg/L) of methylene blue dye; C_e represents the concentration (mg/L) of methylene blue dye at the equilibrium state; V represents the solution volume (L); and m represents the adsorbent mass (g).

To describe the equilibrium nature of the adsorption of the dye on the activated carbon adsorbent, Langmuir and Freundlich adsorption models were used. The Langmuir model assumes that an adsorbate monolayer is adsorbed uniformlu on the adsorbent surface. According to eqn. 2, adsorption only occurs at particular adsorbent sites when it forms a monolayer on the surface.

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

where q_m represents the maximum amount of methylene blue dye absorbed per unit weight of adsorbent (mg/g); C_e represents the concen-tration of methylene blue dye at equilibrium (mg/L); q_e represents the amount of methylene blue dye adsorbed per unit weight of the adsorbent at equilibrium and K_L represents the Langmuir constant (L/mg). The slopes and intercepts of the plots of C_e/q_e *vs.* C_e were used to compute the values of q_m and K_L, respectively.

The adsorption properties of the heterogeneous surface can be described by the Freundlich adsorption isotherm. The empirical equation proposed by Freundlich can be represented by using eqn. 3:

$$\log q_e = \log K_F + \frac{1}{n} \log C_e \tag{3}$$

where C_e represents the concentration of methylene blue dye at equilibrium and q_e represents the amount of methylene blue dye adsorbed per unit weight of the adsorbent at equilibrium; K_F and n represent the Freundlich constant and adsorption intensity, respectively. The linear plots of log q_e *versus* log C_e were used to determine the values of K_F and 1/n (between 0 and 1).

RESULTS AND DISCUSSION

Characterization: The morphological characteristics of LDW, charcoal and activated carbon samples were determined by scanning electron microscopy (Fig. 1). It was found that the porosity on the surface of activated carbon (Fig. 1c) was higher than that on the surface of LDW (Fig. 1a) and charcoal (Fig. 1b). These results indicated that NaCl can be activated by charcoal to act as an adsorbent of activated carbon.

The chemical composition and purity of the activated carbon adsorbent were analyzed by EDX (Oxford, Aztec, UK). The EDX spectra (Fig. 2) showed that various elements were present including carbon (C), oxygen (O), silicon (Si) and sodium (Na), which accounted for 64.4%, 22.5%, 12.6% and 0.5%, respectively. The EDX mapping technique is used to examine the composition and distribution of elements in a sample. The EDX mapping of the obtained activated carbon is shown in Fig. 3 and it was found that all the elements were distributed uniformly.

The FTIR spectrum analysis was conducted to characterize the surface of LDW, charcoal and activated carbon. The FTIR spectra were obtained from FTIR-Bruker, Germany, instrument. Fig. 4 shows the FTIR spectrum of LDW (Fig. 4a), the charcoal sample obtained after pyrolysis at 400 ℃ (Fig. 4b) and the activated carbon obtained by chemical treatment with NaCl followed by pyrolysis at 850 ℃ (Fig. 4c). The FTIR analysis of LDW before treatment with NaCl and before pyro-
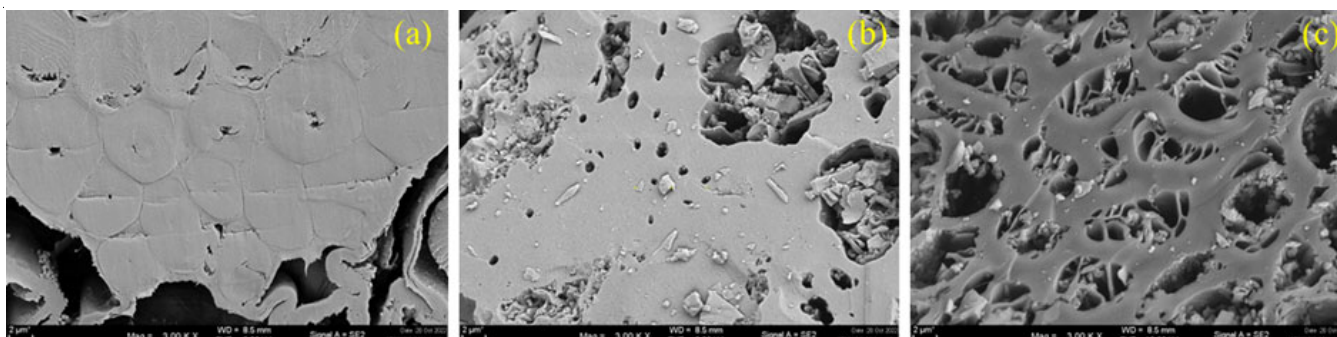
Fig. 1. SEM images of (a) *Lepironia articulata* (Retz.) Domin waste, (b) charcoal and (c) activated carbon
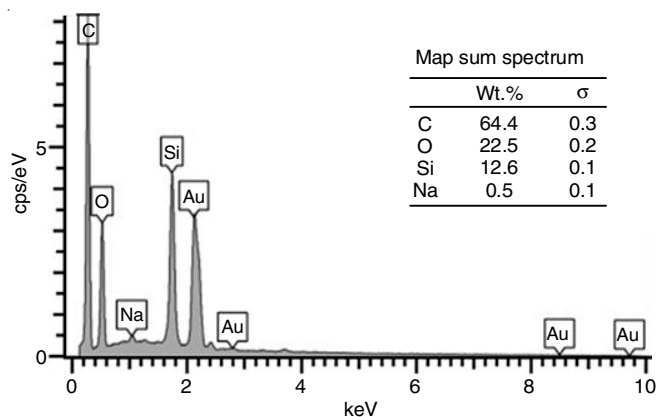

Map sum spectrum		
	Wt.%	σ
C	64.4	0.3
O	22.5	0.2
Si	12.6	0.1
Na	0.5	0.1

Fig. 2. EDX spectra of activated carbon

lysis at 850 ºC showed bands at 3389, 2916, 1645, 1463 and 1018 cm^{-1} belonging to the O-H, C-H, C-O, CH$_2$ and C-O-C groups [27], respectively. When compared the spectra of charcoal after pyrolysis of the sample at 400 ºC (Fig. 4b) to the spectra of activated carbon obtained after treatment with NaCl followed by pyrolysis at 850 ºC (Fig. 4c), the distinctive bands around 1159, 820 and 450 cm^{-1} are due to the -CO, Si-O bending and Si-O rocking, respectively. The distinctive bands were absent in the other regions of the spectra probably because the structure was strongly carbonized.

Adsorption experiments

Effect of dye concentration: The intial concentration of methylene blue dye facilitated the efficient transfer of the dye
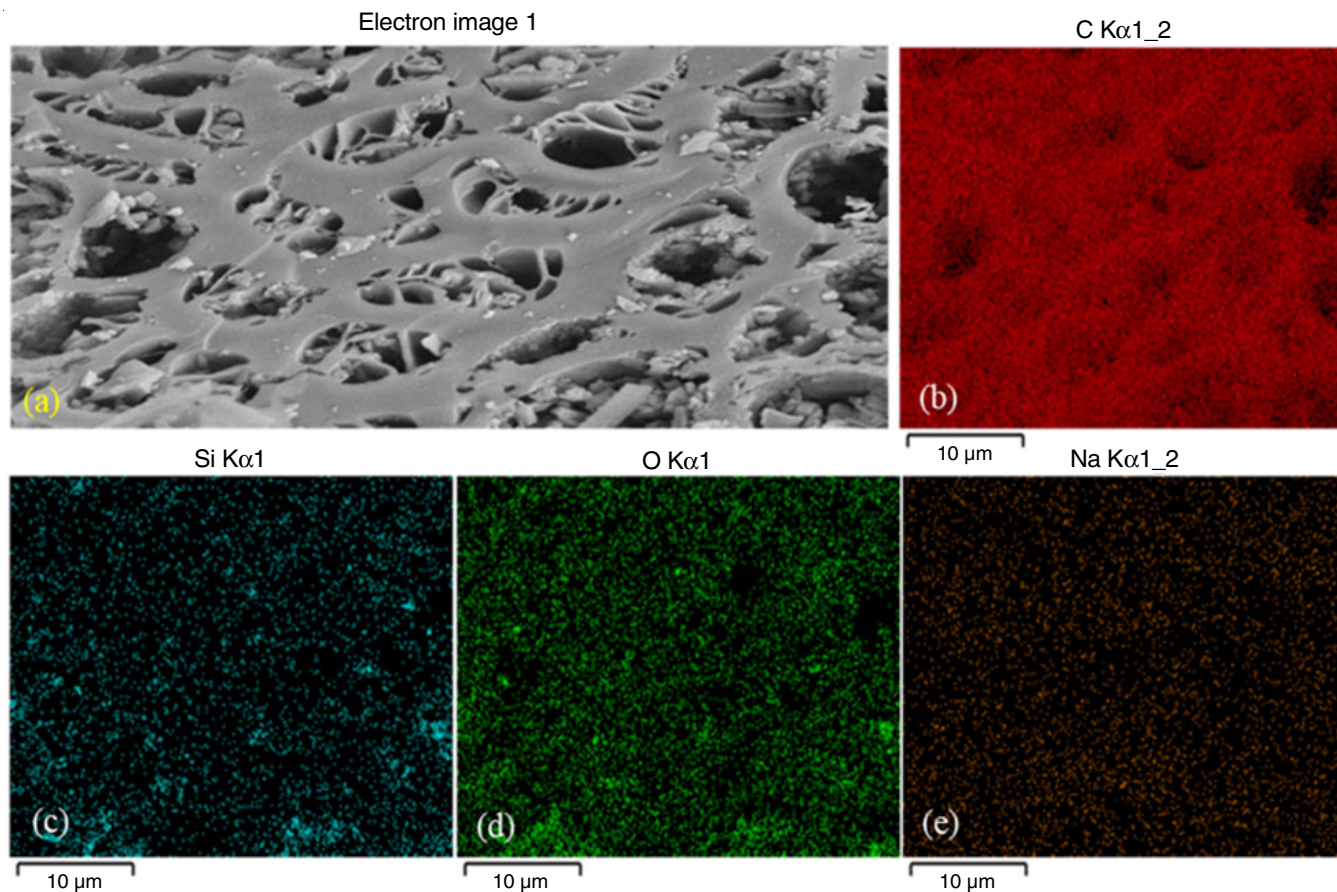

Fig. 3. (a) SEM image and EDX mapping analysis of activated carbon adsorbent (b) carbon, (c) silicon, (d) oxygen and (e) sodium
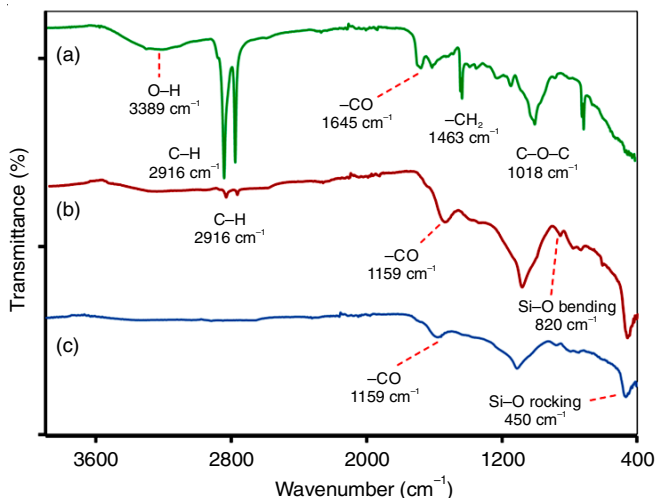
Fig. 4.   FTIR spectra of (a) *Lepironia articulata* (Retz.) Domin waste, (b) charcoal and (c) activated carbon

between the aqueous and solid phase adsorbents by reducing the resistance to mass transfer. The effect of different concentrations of dye (5 to 40 mg/L) on dye removal using activated carbon is shown in Fig. 5. The adsorption capacity increased from 12.50 to 66.25 mg/g. As more sorption sites were present on the surface of activated carbon than the number of methylene blue dye molecules available when the concentration of methylene blue dye was low and most of the methylene blue molecules were adsorbed. The adsorption of a high proportion of methylene blue dye molecules indicated high sorption efficiency. However, a limited number of methylene blue dye molecules available to the activated carbon could lower the sorption capacity. In contrast, at higher concentrations of methylene blue dye, fewer active sorption sites were available on the surface of the activated carbon than the number of methylene blue dye molecules present. This forced the sorbate molecules to compete with one another for the limited number of sorption sites. Thus, extra sorbate molecules could not bind to the surface of the activated carbon and remained free in the solution. Thus, as the concentration of methylene blue dye increased, adsorption capacity increased, but adsorption effectiveness decreased [28]. Thus, the optimal concentration of methylene blue dye was found to be 30 mg/L and this concentration was selected for further measurements.

Effect of adsorbent dosage: The adsorption of the dye in the presence of various amounts of adsorbent was evaluated to determine the highest adsorption with the least amount of adsorbent. The percentage of methylene blue dye removed increased with an increase in the adsorbent dose (0.01 to 0.08 g) (Fig. 6), but the difference in the removal after 0.08 g of adsorbent was negligible. A higher proportion of methylene blue dye was removed as the adsorbent dose-to-methylene blue dye concentration ratio increased. This occurred because a fixed dose of adsorbent could absorb only a certain amount of dye [29]. Therefore, more adsorbent was needed to purify a larger volume of effluent for a specific concentration of dye. The optimal dose of the activated carbon was found to be 0.08 g, which was used for further measurements.
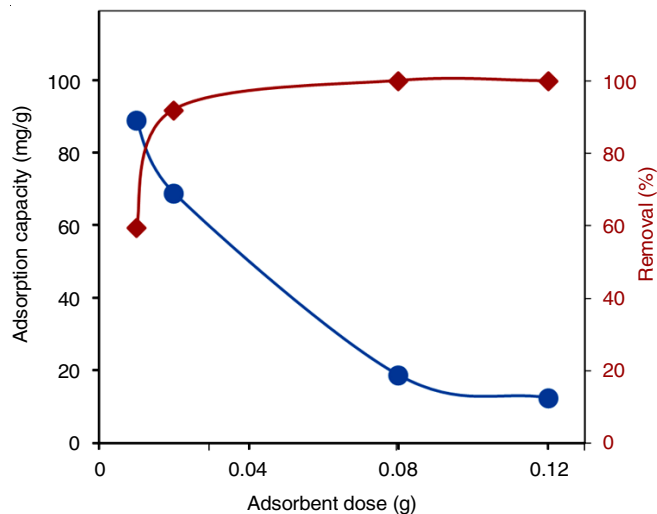
Fig. 6. Effect of adsorbent dose

Effect of contact time: The effect of contact time on the elimination of methylene blue dye is shown in Fig. 7. When activated carbon was used as an adsorbent, 90.39% of dye was removed in 60 min. The equilibrium was reached after 90 min. A large number of empty adsorbent sites at the beginning and a very high solute concentration gradient might have contributed to the shift in the rate of adsorption. At later stages, a decrease in the number of empty sites for the adsorbent and the dye led to a reduction in the adsorption rate. The lower adsor-
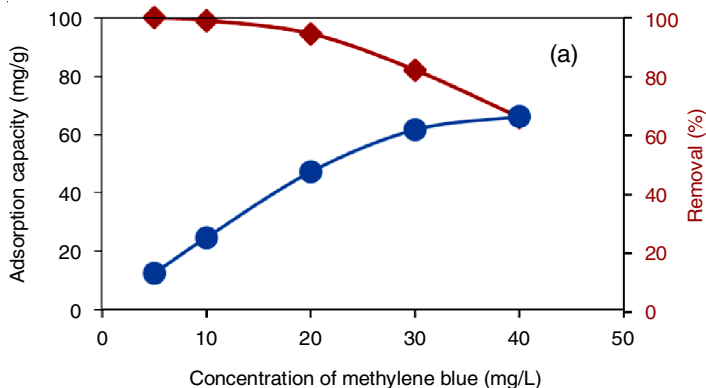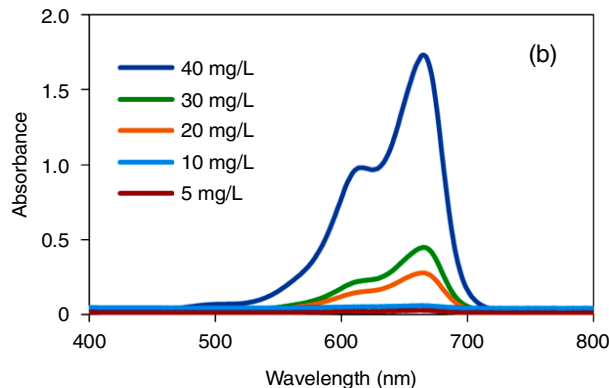

Fig. 5.   Effect of the concentration of the dye on (a) the adsorption capacity and percentage removal of activated carbon, (b) the methylene blue spectrum at different concentrations
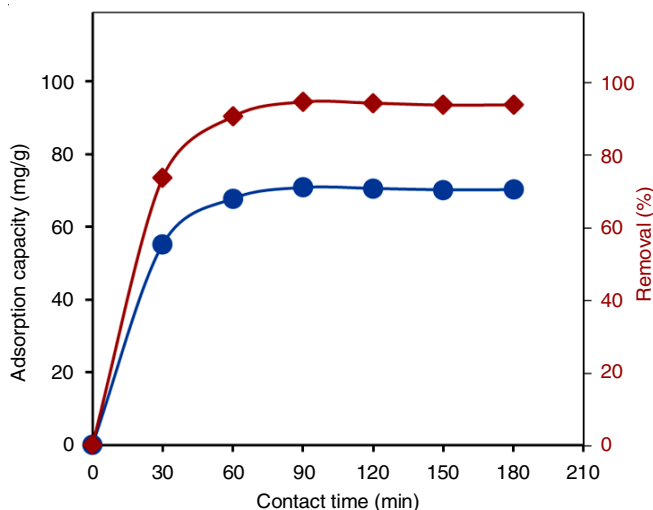
Fig. 7. Effect of contact time on the removal of methylene blue dye

ption rate at the end of the experiment suggested that methylene blue dye might have formed a monolayer on the adsorbent surface. This probably happened since once equilibrium was reached, there were not enough active sites remaining to allow for further uptake, thus preventing further uptake [30]. The optimal contact time was found to be 90 min, which was selected for further experiments.

Adsorption isotherms: For designing any adsorption system, equilibrium adsorption isotherm models are essential. Using linearized forms of Langmuir and Freundlich isotherms, the equilibrium sorption data were analyzed.

The fitted plots of Langmuir adsorption and Freundlich adsorption of methylene blue dye using the activated carbon obtained from *Lepironia articulata* (Retz.) Domin waste (LDW) are shown in Fig. 8. The values of the constants obtained from the Langmuir ($q_m = 69.44$ mg/g; $K_L = 0.02$ L/mg; $R^2 = 0.9978$) and Freundlich adsorption ($K_F = 0.31$ mL/g; $n = 2.49$ and $R^2 = 0.0.9715$) isotherms along with their correlation coefficients (R^2). The results showed that the Langmuir isotherm ($R^2 > 0.99$), which indicated that the adsorbent surface was homogeneous, fit the experimental results for activated carbon adsorbent better than the Freundlich isotherm ($R^2 > 0.97$). The maximum adsorption capacity was 69.44 mg/g. The highest capacity of this dye to bind to various adsorbents is presented in Table-1 for comparison. By comparing the activated carbon material to other

TABLE-1
A COMPARISON OF METHYLENE BLUE MAXIMUM
ADSORPTION CAPACITY (q_m) OF ONTO ACTIVATED
CARBON PREPARED FROM VARIOUS PRECURSORS

| Adsorbent	q_m (mg/g)	Ref.
Cumin (*Nigella sativa* L.) seeds	15.47	[31]
Cashew nut shell	68.72	[32]
Coconut coir dust	14.36	[33]
Sago waste	4.51	[34]
Banana pith	4.67	[34]
Yellow passion fruit waste	44.70	[35]
Spent coffee grounds	18.70	[36]
Waste orange and lemon peels	33.00	[37]
Acacia erioloba seed pods	95.42	[38]
Nauclea diderrichii agricultural waste	35.09	[39]
Lepironia articulata (Retz.) domin waste	69.44	This work
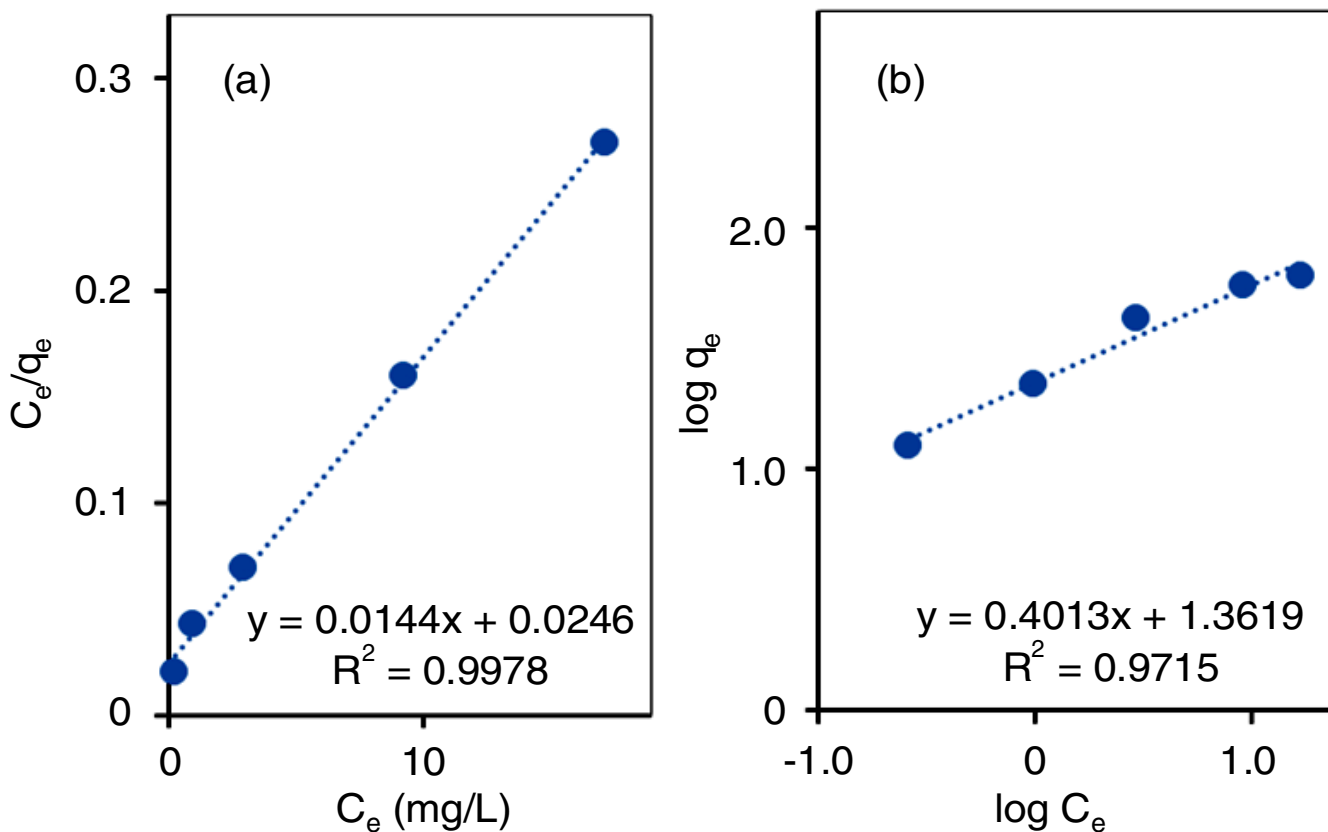
Fig. 8. Linear plots of (a) Langmuir and (b) Freundlich isotherm models for activated carbon adsorbents

adsorbents, it was found that it had a high adsorption capacity and could effectively remove methylene blue dye from the aqueous solution.

Environmental application: The wastewater from the local fabric dyeing in Nakhon Si Thammarat Province, Thailand, was treated with the obtained activated carbon adsorbent to remove dyes of different colours. The present results showed that when the percentage of elimination is greater than 60%, the activated carbon adsorbent can be used for treating waste-water containing various dye contaminants (Table-2).

TABLE-2
PERCENTAGE OF DYE REMOVED AFTER TREATMENT WITH THE ACTIVATED CARBON ADSORBENT

Dye type	Dye concentration adsorption (mg/L)		Dye removal (%)
	Before	After	
Dark blue dye	30.0	8.6	71.3
Red dye	30.0	11.2	62.7
Pink dye	30.0	10.1	66.3
Sky blue dye	30.0	9.5	68.4
Yellow dye	30.0	8.4	72.0

Conclusion

Activated carbon from *Lepironia articulata* (Retz.) Domin waste was investigated in this study and found to be an efficient adsorbent for the removal of methylene blue dye from waste-water. The optimized parameters such as the concentration of the methylene blue dye, the adsorbent dosage and the contact time were found to be 30 mg/L, 0.02 g and 90 min, respectively. The Langmuir isotherm proved to be an effective model for evaluating the adsorption process. The sample had a maximum adsorption capacity of 69.44 mg/g and a removal percentage of greater than 90%. When applied to real wastewater samples, this approach yielded samples with a dye removal percentage greater than 60% for a variety of colour dyes. Present findings indicated that the activated carbon produced from *Lepironia articulata* (Retz.) Domin waste performed well and might be used as an adsorbent for removing the toxic dyes from the wastewater.

CONFLICT OF INTEREST

The authors declare that there is no conflict of interests regarding the publication of this article.

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