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Lignocellulosic Biomass Waste Adsorbent for Removal of Dye from Aqueous Solution

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Abstract

Date Palm Tree Fiber (DPTF) is a renewable raw material which is considered a biomass waste excess of date farms and manufactures. This biomass was successfully utilized as solid phase extractor (SPE) for removal of methylene Blue dye (MB) from polluted water. The chemical composition and the surface morphology of the SPE was critically assigned based on FT-IR, and FE-SEM. In batch separation mode, the impact of several analytical parameters e.g. contact time, weight of biomass, solution pH and initial concentration on the MB dye uptake from aqueous media by DPTF solid adsorbent was critically studied. At the optimized parameters, good adsorption capacity ($q_{e,exp}$ =9.153 mg/g) at pH 6 was achieved. The data of MB uptake were subjected to Langmuir and Freundlich to assign the most probable retention mechanism. The adsorption fitted well with Langmuir isotherm models q_m 648 mg/g. The data were fitted well with pseudo- second order model as supported from the $q_{e,cal}$ (9.225 mg/g) value. Excellent extraction (99%) of MB dye was achieved from the environmental water samples. The DPTF solid sorbent is a non-toxic and renewable raw material of lignocellulosic biomass has a potential to uptake MB dye from wastewater.

Keywords: agri-food waste, adsorption dye, recycled materials, environmental water samples

1. Introduction

Wastewater samples are frequently released into water streams with great harmful effects to the aquatic environment and to human health. Industries that use dyes to convey a color they desire to their products such as food, wood, rubber, textile, plastics, and cosmetics, pharmaceutical industries create too much contamination in the aquatic environment (Alrumman *et al.*, 2016). The dye-contained discharges are expected to be a considerable concern due to the harmful impacts of dye in several elements of life cycle (Alrumman *et al.*, 2016). The toxicological and aesthetical issues accompanied with the release of dyes into the wastewater are major causes of these concerns. Since water is the most important element of life on earth. Thus, the problem of water purification and providing effective and cheap solutions are very important. Methylene blue (MB) dye is one of the extensive raw materials in dye usually applied for cotton and silk paints. The most harmful side effects of MB dye may generate are eye burns, breathing difficulties, nausea, ejaculating, extensive sweating, confusion, and methemoglobinemia (Senthilkumaar *et al.*, 2005).

A variety of methods have been applied to extract dyes from wastewater; such as flocculation, adsorption, oxidation, electrolysis, biodegradation, ion exchange, and optical catalysis (Kubra *et al.*, 2021) . Among numerous methods, adsorption is an efficient extraction procedure for a broad diversity of applications. Adsorption is a procedure by which a solid adsorbent draws in a component in water to its surface and create a bond through a physical or chemical bond, as a result extracting the component from the liquid stage. It is currently known as an efficient and cost-effective procedure for extracting both raw and non-raw materials from wastewaters (Gabal *et al.*, 2014; Alhogbi, 2017; Hu et al., 2020; Roa *et al.*, 2021).

In addition, biomass produced from agricultural wastes (in millions of tons per year), is an effective substance serving as a solid phase extractor of adsorption technology since the agriculture waste components are cellulose, hemicelluloses, silica, fat, proteins, lignin, hydrocarbons, starch, simple sugar, and water; most of these materials have different functional groups with potential adsorption for various contaminants (Eloy de Souza *et al.*, 2020). Therefore, lignocelluloses biomass adsorbent is documented as the most promising fundamental approach in wastewater treatment because of its manageable pore structure, vast porous surface area, low reactivity to

acidity/basicity, and thermo-stability (Alhogbi, 2017). Adsorption techniques have been widely studied regarding effectivity, simplicity, and economically for the removal of organic and inorganic pollutants from wastewater (Franca *et al.*, 2009; Warade *et al.*, 2016; Yi *et al.*, 2016; Yaseen & Scholz, 2018; Alhogbi *et al.*, 2019; El-Refaie *et al.*, 2022).

Nowadays, the ecological and economic issues commitments are the cultivating locals from side-effects of agricultural wastes. In this area, the recycle economy plans to decrease waste and utilize the agricultural waste by applying eco-nomically feasible processes to rise their qualities. Herein the Date Palm Tree Fiber (DPTF), a harmless waste excess from date farms and manufactures were investigated as a natural solid phase extractor for the adsorbent of Methylene Blue dye (MB) from polluted water. The impact of pH, weight of biomass, initial concentration, and contact time on the rate of removal of (MB) dye were studied. The data used the adsorption isotherms and Kinetic models to review the adsorption process purpose.

2. Methods and Materials

2.1 Preparation, Characterization, and Morphology of (DPTF) Adsorbent

Date Palm Tree Fiber (DPTF) was cleaned from dust by washing various times using distilled water and kept drying at room temperature for few days, then it was electrically grinded. DPTF was sieved to 0.85 mm particle size and stored into plastic bags. The DPTF characterized and surface morphology was studied using Fourier Transform Infra-red spectroscopy (FT-IR) and Field Emission Scanning Microscopy/Energy Dispersive X-ray (FE-SEM- JEOL JSM 6360LA, Japan). The FE-SEM analysis was carried out at room temperature with accelerating voltage of 20 kV.

2.2 Batch Experiment

Batch experiment was used to study the effectiveness of DPTF for removal the Methylene blue (MB) from wastewater. The effect of different environmental factor such as, adsorbent mass, initial concentration, contact time, and pH were studied. A stock solution (100 µg/mL) of MB sample was prepared in deionized water. A measured amount of (5.0 mg to 50.0 mg) the DPTF was added to the dye solutions of 25 mL in conical flasks placed on a shaker (GFL Shakers, 3006) at agitation speed 250 rpm at room temperature for (2 and 90 mins). The pH of the metal ion solution was adjusted by adding a few drops of 1 M of NaOH and 1 M of HCl for pH (2.0 to 10) a digital pH meter (Mettler Toledo MP220). The metal solution was filtered by using filter paper Whatman No. 2 and then taken for further analysis. All determinations were performed in triplicates per experiment the concentration in the solution was measured by a UV visible spectrophotometer (UV-1650PC, SHIMADZU) two 15-watt lamps (Philips Model) at λ_{max} of 664 nm and the average values were reported. The amount of MB dye retained per unit mass q_e (mg/g) and the percentage removal were calculated using the following equations:

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

$$\%\ Removal = \frac{C_i - C_e}{C_i} \tag{2}$$

where C_i and C_e are the initial and equilibrium concentrations of dye solution (mg/L), respectively. V is the volume of dye solution (L), and m is the amount of the DPTF (mg).

2.3 Sorption Isotherm and Kinetic Study

Langmuir and Freundlich models for MB retention were expressed by the following equations, respectively:

$$\frac{C_e}{q_e} = \frac{1}{K_l\ q_m} + \left(\frac{1}{q_m}\right)\ C_e \tag{3}$$

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

Whereas the kinetic study involving pseudo-first order and pseudo second order models of MB uptake were also expressed by the following equations, respectively:

$$\log(q_e - q_t) = \log q_e - \frac{k_1}{2.303}t \tag{5}$$

$$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{1}{q_e}t \tag{6}$$

where: q_e – amount of adsorbate adsorbed per unit weight of adsorbent at equilibrium, mg/g, q_m – Langmuir adsorption capacity, (mg/g), k_L – Langmuir constant, ((L/mg), k_F – equilibrium Freundlich constant indicative of adsorption capacity, $((mg/g)(L/mg)^{1/n}$, 1/n – measuring the adsorption intensity, k_1 - pseudo-first-order rate , t - contact time, (min) and k_2 - pseudo–second-order constant rate, (g/ mg min).

3. Result and Discussion

3.1 Characterization Studies of DPTF

Scanning electron microscopy (SEM) is one of the most versatile and well-known techniques applied to investigate the surface morphology of the solid adsorbent. The SEM exhibited that the DPTF was composed of large layered coarse irregular and heterogeneous particles as shown in Fig.1 (a). FT-IR spectra of the MB, DPTF, and DPTF-MB (Fig. 1 b) showed the carboxylic, hydroxyl, and amine on the adsorbent's surface, in addition to the existence of the groups in the lignin, cellulose, or amine (NH_2) and starch groups. The functional groups were empowered by natural adsorbents through different processes such as adsorption, ion exchange, and complexation to remove dyes from contaminated water (Roa *et al.*, 2021; Eloy de Souza *et al.*, 2020; Alhogbi *et al.*, 2019). The hydroxyl (OH) functional groups were observed for stretching and bending vibration bands at 3470 cm^{-1} and 1650 cm^{-1}, respectively. Stretching vibrations band at 1440 cm^{-1} of carboxyl group (COOH). The spectrum after adsorption DPTF-MB shows the strength of adsorption peak 3470 cm^{-1} and 1140 cm^{-1} decreased which means it was contributed to the adsorption process. The peak at 1050 cm^{-1} was assigned to the stretching C-O bond in cellulosic glucose ring. The spectra in Fig.1 (b) shows the band at 910 cm^{-1} was assigned to N-H wag in MB dye.

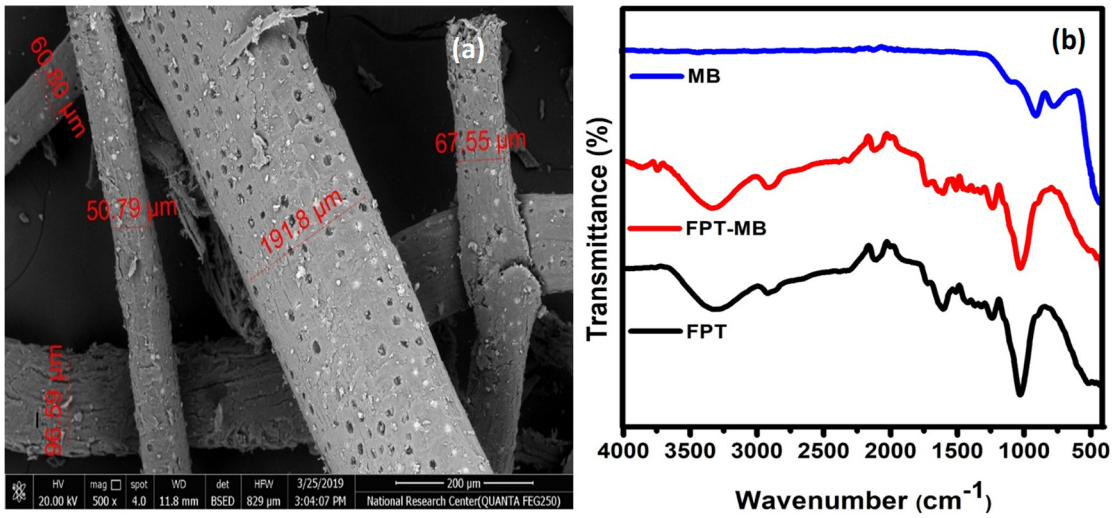

Figure 1. Characterization of (DPTF) (a) SEM micrograph of the adsorbent material amplified 7,500 and (b) FTIR spectra

3.2 Analytical Parameters

3.2.1 Impact of Adsorbent Dosage

Impact of adsorbent dosage showed various MB dye percentage removal with difference of (5.0 – 50 mg) DPTF dosage in 25 mL MB solutions of 5.0 mg/L is introduced in Fig. 2 (a). The removal efficiency increases from 86.1 % to 99.7 % on increasing the adsorbent dosage from 5.0 mg to 50 mg. The removal efficiency increases with adsorbent dosage increase which apparently might be due to the availability of the active sites vacant on the surface of the adsorbent. At dosage of 20 mg the maximum percentage removal of MB was about 99.7 %, thus, it was selected as optimum dosage for additional studies.

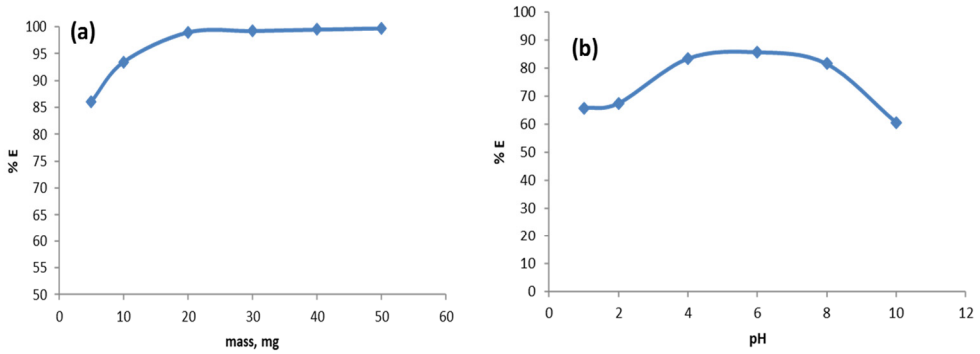

Figure 2: The different influence of MB dye adsorbed onto DPTF (a) adsorbent dosage, (b) solution pH, at T= 298 K, V = 25 mL, C = 5.0 mg/L, t = 90 min.

3.2.2 Impact of pH

The impact of pH apparently influencing on adsorption, was examined in a pH range (1 – 11). Fig. 2 (b) shows the influence of pH on the adsorption of MB into DPTF. The data found that at pH 2 a low percentage of removal was given at 65.0 %. Ordinarily, in an acidic medium, there are more positive charges would be on the surface of the adsorbent, that cause an electrostatic repulsion between the positive charge on the binding site of the DPTF and cation MB dye. When increasing the pH to 6, it led to an increase of the removal efficiency to 85.6 %, owing to a significant increase in electrostatic force between the adsorbent and the adsorbate (Mohammed *et al.*, 2014). While increasing the pH 8 to 11 the percentage removal was decreased to 75.0 %, and 65.0 %, respectively. At the alkaline medium the adsorption decreases because OH^- ions were formed [involved] which tend to form deposits on the adsorbate MB dye and adsorbents DPTF. The highest adsorption percentage of cationic MB occur was 85.6% at pH 6 which indicated that the surface charge of the DPTF expresses negative charge owing deprotonation in the present of hydroxyl ion (Hu *et al.*, 2020).

3.3 Adsorption Isotherm

The impact of the initial concentration of the adsorbate plays an important role in determining the amount of adsorbate adsorbed. Fig. 3 (a) shows the impact of initial concentration of MB dye from 2.0 mg/L to 90 mg/L found that the adsorption capacity was increased from 8.4 mg/g to 9.953 mg/g on DPTF. The adsorption process led to an increase the amount of the adsorption capacity within an increase in the initial concentration of dyes. This result may be attributed to the availability of large number of MB dye molecules in the process between the solid phase and the liquid phase generate a high driven force to surpass the resistance of mass transfer molecules (Chan *et al.*, 2016). The adsorption capacity reach equilibrium of MB from aqueous solution by using DPTF was found to be 9.86 mg/g at MB concentration 30 mg/L. Regarding to the maximum adsorption, Safa & Bhatti (2011) stated that the amount of a maximum adsorption capacity has a strong interaction between the initial dye concentration and the amount of the adsorbent dose.

To examine the interaction of adsorbate molecules concentration and adsorbent surface, two applicable models, the Langmuir and Freundlich isotherms were chosen to clarify interaction of MB dye with DPTF, it was shown in Fig. 3 (b, c). In Fig. 3 (b) exhibits the Langmuir isotherm by plotting the C_e/q_e versus C_e meanwhile the linear correlation coefficient value (R^2) signifies the model, it has high R^2 value of MB 0.9986 in the Langmuir isotherm. In Fig. 3 (c) shows the R^2 value in Freundlich isotherm was 0.8848. The data indicated that the Langmuir adsorption isotherms model was best fitted to examine the interaction (Langmuir, 918). Table 1 illustrated the resulted data of a maximum monolayer capacity using the Langmuir model (q_m) of MB dye was 648 mg/g by DPTF, (Aly, 2014; Somaia, 2017). Furthermore, the high value of the constant of K_L was 1.8×10^{-4} L/mg for MB dye, which indicates the strong bonding of dyes onto DPTF (Hassan, & Elhadidy, 2017). Similar studies were reported on the adsorption of MB dye onto *Empty Fruits Bunch* activated carbon (Egbosiuba, 2020).

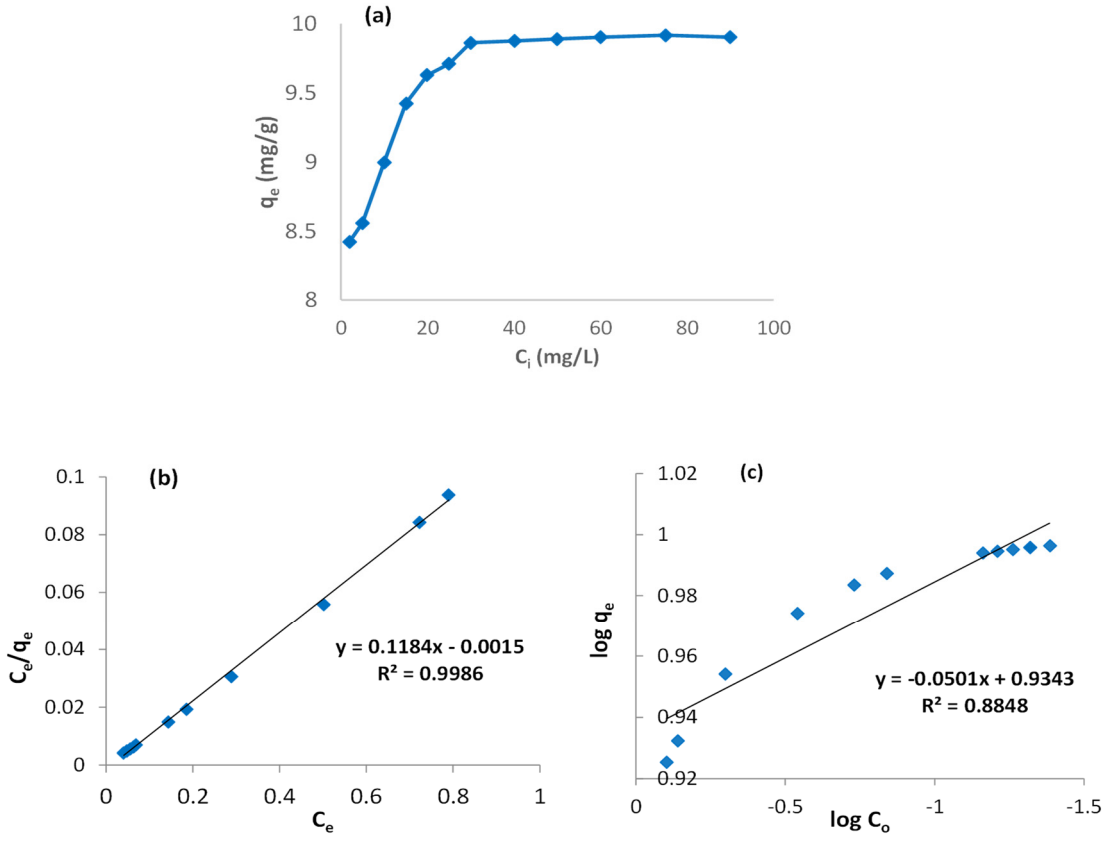

Figure 3: (a) The impact of initial concentration Ci, (b) Langmuir, and (c) Freundlich Linearized isotherms models for the adsorption of MB dye by DPTF at T= 298 K, V = 100 mL, m = 20.0 mg, pH= 6, t = 90.

Table 1: Adsorption isotherm parameters for MB dye adsorbed onto DPTF at 298 K

	q_m (mg/g)	R^2	K_l (L/mg)	K_f ((mg/g) $(L/mg)^{1/n}$	1/n
Langmuir	648	0.999	1.8×10-4		
Freundlich		0.885		8.596	19.954

3.4 Adsorption Kinetic Models

Impact of contact time on the adsorption of MB onto DPTF at 298 K is presented in Fig. 4 (a). The adsorption of MB onto DPTF increases rapidly in the first 30 mins this might be due to available of vacancy of binding sites, then the adsorption rate became slower and eventually equilibrium was attained at 75 mins, because the most binding sites were occupied (Franca, 2009). Furthermore, the impact of contact time finding was performed to the kinetics model provides substantial evidence to identify the adsorption mechanisms by establishing a linear pseudo-first order (Eq. 5) and pseudo-second order models (Eq. 6) presented in Fig. 4 (b, c). The adsorption kinetics parameters are illustrated in Table 2. Using the pseudo second order equation by plot of t/q_t versus t for yields a good straight line that obtained coefficient value ($R^2 > 0.99$). Obviously, the theoretical amount of adsorbate adsorbed at equilibrium ($q_{e,cal}$) value was 9.225 mg/g for MB dye was comparable to the experimental ($q_{e,exp}$) value 9.153 mg/g. Thus, kinetic studies of adsorption revealed that the adsorption process followed a pseudo second order kinetic model. The data in this study is in the same way as the results reported on the adsorption of MB dye onto *Waste tea* activated carbon, and *Empty Fruits Bunch* activated carbon (Egbosiuba, 2020).

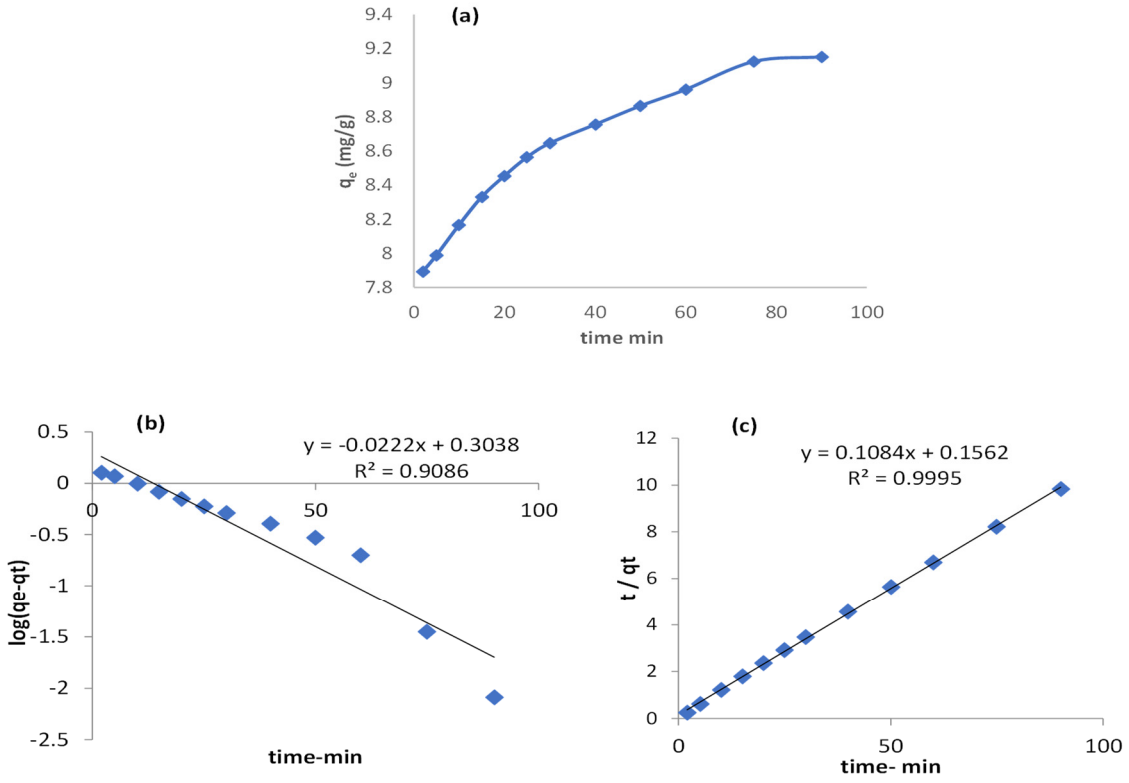

Figure 4: (a) The impacts of contact time, (b) Pseudo-first order, and (c) Pseudo-second order Kinetics models for the adsorption of MB dye by DPTF at T= 298 K, V=100 mL, m= 20.0 mg, C= 50.0 mg/L, pH= 6.

Table 2. Adsorption kinetics models for MB dye adsorbed onto DPTF at 298 K

	q_{exp} (mg/g)	q_{cal} (mg/g)	R^2	K_1 (min^{-1})	K_2 (g/ mg min)
Pseudo - first order		2.013	0.908	0.051	
Pseudo- second order	9.153	9.225	0.999		0.075

3.5 Environmental Application

The DPTF has been regarded as an effective sorption of MB dye from aqueous solution. Therefore, to investigate the effectiveness and firm in the environmental samples such as seawater and tap water. The batch method of 30 mg/L MB dye concentration was added to the environmental water. The result exhibited that, the DPTF uptake of MB dye adsorption percentage was (99.5 %, 99.3 %) in the tap water and seawater, respectively. Although, there was no significant difference of removal percentage, seawater has high concentration of dissolved inorganic ions than tap water. The application in environmental samples demonstrated a good dye adsorption, that display the DPTF adsorbent's efficiency has the potential of application purpose for environmental water remediation in the future.

4. Conclusion and Future Perspectives

The DPTF has applied as an adsorbent material factor that has been considered including the adsorbent dose, pH, initial concentration, and contact time. The DPTF surface morphology was coarse irregular particles proved by SEM. In the components of fibrous natural biomass, the functional groups identified by FT-IR that was carboxylic, hydroxyl, and amine. The maximum percentage removal of MB was about 99 %, at the dosage of 20 mg at pH 6 and the adsorption capacity obtained was $q_{e,exp}$ 9.86 mg/g of 30.0 mg/L MB dye concentration after 90 mins. Adsorption isotherms, and kinetic models were carried out for the calculation of adsorption process. The isotherm

data demonstrated that adsorption process provides a better fit to the Langmuir isotherm due to R^2 values akin to one. The adsorption rate process tends to obey a pseudo second order kinetic model while the mathematical adsorption capacity was $q_{e,cal}$ 9.225 mg/g, indicates that the rate controlling step might be ion exchange or electrostatic force between the active functional groups in DPTF and MB dye. The effective of DPTF on the seawater and tap water samples for removal MB Dye was high at 99 % percent. The result introduced in this investigation indicates that DPTF presents extraordinary potential as a reasonable and effectively accessible alternative adsorbent for the removal of cationic dyes from wastewater. This project will continue to investigate the increase of adsorption capacity by modifications of the DPTF surface efficiency for water remediation. Design experiment is highly recommended to consider the interaction between parameters.

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