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Article

Investigation of a Palm Fronds as Affordable Adsorbent for Eliminate Dye from Wastewater

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Abstract: The textile industry discharges large amounts of dye-contaminated water annually to environment, which is harmful to our environment and aquatic life. This study investigates the utilization of palm frond (PF) an inexpensive and largely available agricultural waste material to eliminate methylene blue (MB) present in effluent. To solve this problem, PF were harvested, and characterized by SEM, EDS and FTIR.

Batch experiments were then conducted using several main variables which are: pH, time of contact, amount of adsorbent and initial concentration from dye. Removal efficiency was found to be as high as 97.28% at an equilibrium time of 60 min and 30°C under alkaline conditions (pH 12). Adsorption isotherms fitted laboriously to different models were analyzed, leading to very close correlation fits (Langmuir $R^2 = 0.9855$; Freundlich $R^2 = 0.9919$; Temkin $R^2 = 0.8826$), while neither isotherm adequately describes the data except for the Freundlich model indicating a heterogeneous adsorptive surface in these experiments across our experimental range of concentrations tested. Kinetic analysis showed that it corresponds to a model of pseudo second order, which chemisorption represent the major rate controlling process. These results show that palm fronds are a potent, inexpensive, and locally available adsorbent for remediation of contaminated dye wastewater.

Keywords: Adsorption, Palm Fronds, Methylene Blue, Wastewater treatment, Kinetics

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1. Introduction

The explosive expansion of industrialization has grown to be a significant supply of the discharge of wastewater, including numerous organic contaminants, especially synthetic dyes. In addition to using a large amount of fresh water resources, the textile industry bears a large amount of synthetic colourants, making it one of the biggest contaminators on earth. Those dyes are resistant to biological degradation [1] and often contaminate the environment where they can cause serious deterioration of such as reduction of light penetration, disturbance for aquatic ecosystems like lakes and rivers, and potential toxicity for living organisms [2]. Cationic dyes, such as MB are utilized in a wide range of applications, including the textile and other industries. Although it is extensively employed, the discharge into water bodies may present environmental and health dangers such as irritation and adverse effects on aquatic organisms. This is the reason, such dyes should be removed from wastewater prior to its discharge [2], [3].

Among them, many treatments have been established for dye removal such as: membrane filtration [4], ion exchange [5], electrocoagulation [6] and advanced oxidation processes [7], and the like. Many of these methods have obstacles, such as energy consumption, high operating costs, or the by-product formation and release of secondary pollutants. On the other hand, adsorption has become one of the most simple and efficient method with relatively high efficiency, simplicity in operation and capability of removing dyes even traces [8]. The high efficacy of activated carbon has led to its widespread use as an adsorbent. However, regeneration challenges and high price limit its practicality [9]. Thus, there is growing emphasis given to local, cost-effective and eco-friendly substitutes. These substrates are abundantly available, biodegradable, and cost-effective [10], making agricultural wastes and natural biomaterials especially attractive.

The palm fronds are easily available in countries such as Iraq, where abundant number of date palm trees grow and useful wastes from agriculture (by a large part) remain unused. Carboxyl and hydroxyl groups are also present in these materials, which can also play a significant role during adsorption. In this study, palm fronds are investigated as potentially useful and affordable adsorbents for MB elimination from wastewater. SEM, EDS and FTIR were employed to characterize the adsorbent which has been prepared from local raw materials. In order to better understand the mechanism of adsorption, batch experiments were undertaken to evaluate the impact parameters. Adsorption isotherm and kinetic analysis were applied jointly to determine the effect of parameters on adsorption.

2. Materials and Method

Adsorbent Preparation

PF were harvested from a nearby agricultural area in Babylon City, Iraq. Initially, the raw material is washed in hot distillate water and after that in cold distillate water to eliminate dust and other impurities. Post cleaning, the samples were oven-dried at 333 K overnight to eliminate the moisture. Dry samples are sieved and ground. No large particles were detected in the powder. The grain sizes obtained were uniform and also about 150 micron. The dry adsorbent was packaged in hermetically sealed tubes for subsequent usage.

MB Solution Elaboration

The model pollutant was the synthetic MB dye at molecular weight = 319.86 g/mol and structure $C_{16}H_{18}N_3SCl$. Standard solution of MB (1000 mg/L) was prepared by dissolving one gram of MB in one liter of distillate water. Dilution yielded working solutions of the different concentrations. For pH adjustment, 0.1 N HCl or 0.1 N NaOH were employed. All chemicals are analytical grade.

Adsorption Analysis

Batch experimentation was carried out to check the effectiveness of PF in removing MB from effluent. Each experiment used 100 mL of MB solution at a defined concentration (10–80 mg/L) and a different amount of adsorbent (1–5 g/L). During the experiments, the pH was adjusted between 3–12 at 30°C.

A magnetic stirrer was used to stir the mixture at 300 rpm to ensure adequate contact between the adsorbent and dye molecules. A series of samples was collected at various times (5 to 60 minutes) and filtration was used to separate the adsorbent. At 664 nm, a UV/Vis spectrophotometer was used to check the residual dye solution.

The adsorption performance was evaluated by determining capacity of adsorption (q, mg/g) and removal efficiency (RE, %). These were determined from the initial (C_i) and final concentration (C_f), volume of sample in L (V), and mass of adsorbent (W) in gm.

$$q = (C_i - C_f)\, V / W \tag{1}$$

$$\%\ Removal\ Efficiency\ (RE) = (C_i - C_f)/C_i * 100 \tag{2}$$

Characterization of the Adsorbent

Several characterization techniques were employed for the analysis of physical and chemical properties of prepared adsorbent. Morphology of PF surface was analyzed using SEM (scanning electron microscopy), and elements composition were analyzed with EDS (energy dispersive X-ray spectroscopy). A Fourier transform infrared spectrometer (FTIR) was used to detect the functional group of surfaces before and after adsorption. These analyses clarify the adsorption mechanism as well as the PF-MB interaction.

3. Results and Discussion

pH Influence

Solution pH is an effective parameter to influence dye molecules adsorption onto adsorbent surfaces. It controls molecular ionization by altering the adsorbent surface charge. A pH value of 3–12, 1 g/L PF dose, 20 mg/L aquatic concentration, and 30-minute contact time were applied to evaluate the removal of MB. It was observed that maximum removal efficiency increased with increasing pH and showed significant difference up to pH 12 as indicated in Figure 1a.

At a low pH, due to excessive hydrogen ions (H^+) that compete with molecules of dye on adsorption spots, efficiency is decreased. Additionally, a cationic dye is repelled by the positively surface charged through electrostatic repulsion. As pH increases, the adsorbent surface will be negatively charged and electrostatic attraction with molecules of MB present in the solution increases, providing a better adsorption [8].

Effect of Adsorbent Dosage

Varying counts of PF from 1 to 5 g/L, is tested the efficacies of adsorbent dosage as specified in Figure 1b by fixing pH (12), time (30 min), and concentration of MB (20 mg/L). As indicated by Figure 1b, removal efficiency became higher with increasing PF amount because more sites of adsorption were created and surface area was increased [11]. As the improvement became less pronounced beyond an optimal dose of 4 g/L. This is most likely because of particulate aggregation, which reduces effective surface-area and hinders access to active sites [8].

Effect of Time

According to Figure 1c, equilibrium time was determined over a contact time range of 5–60 min (20 mg/L dye concentration, PF dose 4 g/L and pH 12). During the first 30 minutes, adsorption was rapid and removal efficiency increased. Possible reason for this is that there are many active spots available.

Over time, though, the speed started to decline as many of those sites became saturated. After nearly 60 min of contact time, equilibrium was achieved with the maximum removal efficiency reaching out to be 97.28%. There were few significant changes beyond this cut-off.

Influence of the Initial MB Concentration

Initial MB concentration (10–80 mg/L) effect was also investigated and represented by using Figure 1d (PF dose 4g/L, time 60 min, and pH 12). The findings have shown that the removal efficiency decreased with a higher concentration. When not enough is adsorbed, there are more sites to bond with because of lower concentrations - so higher efficiency in those cases. However, when dye molecules are concentrated, the number of available sites exceeds that of dye molecules, resulting in a decrease in removal efficacy.

at higher concentrations the number of dye molecules exceeds that of sites available and removal efficiency degrades. Besides, at higher concentration, dye aggregation can also limit diffusion to the adsorbent surface [12], [13].

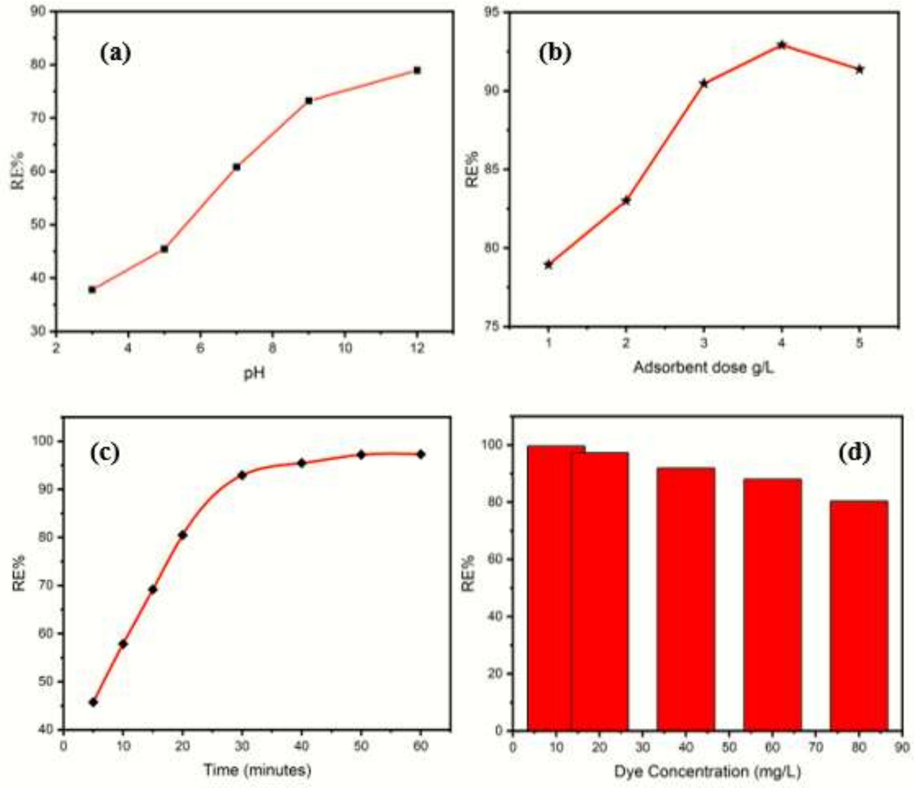


Figure 1 Effect of factors (a) pH solution, (b) PF dose, (c) reaction time, & (d) concentration of dye

Adsorption Kinetic

MB adsorption onto PF kinetics were examined with PFO (pseudo-first order) [11], PSO (pseudo-second order) [14], and Weber and Morres [15] models at pH 12, PF amount 4 g/L, and 20 mg/L MB concentration. The findings appearing in Table 1 and Figure 2 indicate that PFO model ($R^2 = 0.9433$) fits poorly with experimental data compared to PSO model ($R^2 = 0.9964$) at different reaction times for modeling adsorption kinetics on the adsorbent.

Moreover, the adsorption capacity calculated (q_e,cal) for PSO model (5.626 mg/g) is higher than experimental (q_e,exp) value (4.864 mg/g), indicating that the process of chemisorption mechanism as dominated mode. The Weber and Morres [14], [16] intra-particle diffusion model indicates that adsorption involves several resistances in a multistage operation where pore diffusion and adsorption surface play a major role. Since the drawing didn't pass across origin point, diffusion of intra-particle isn't a rate controlling process only [14], [17].

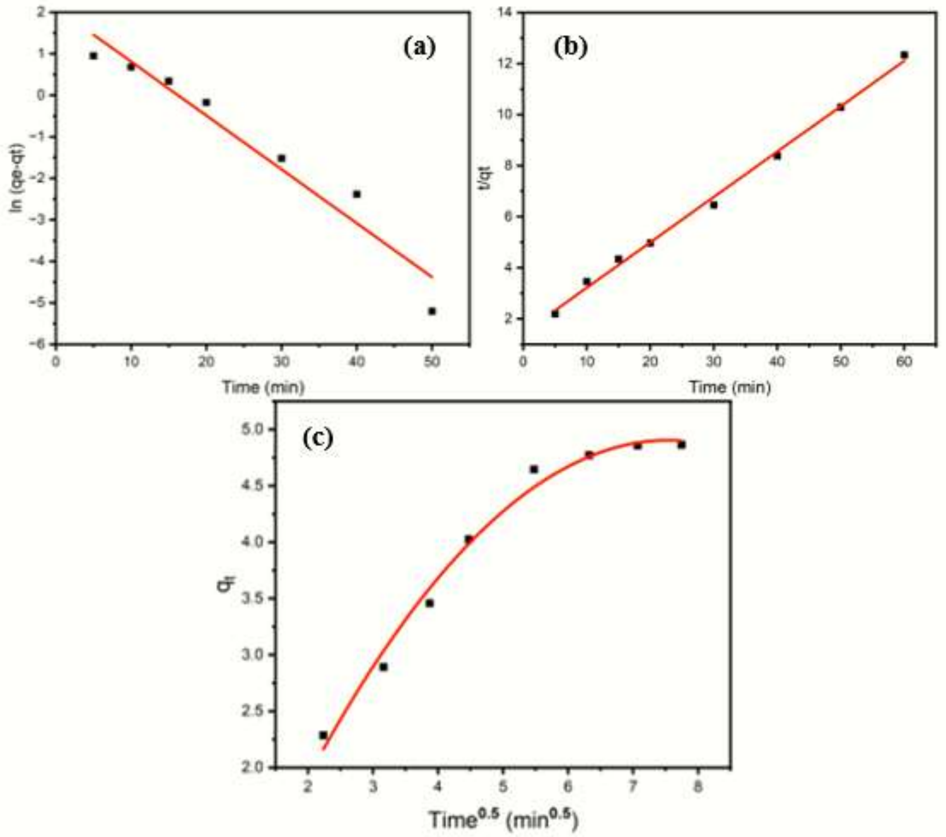
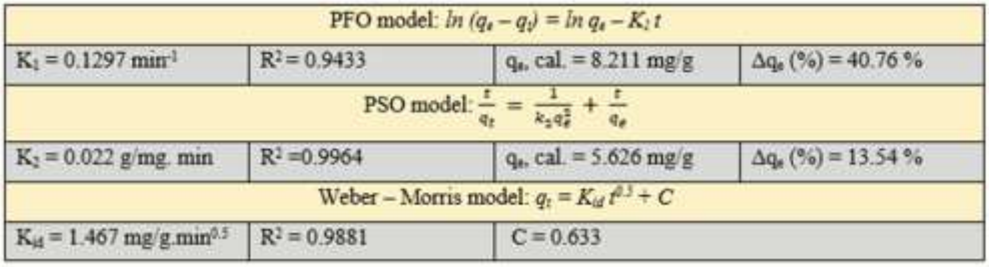


Figure 2 MB Adsorption Kinetics: (a) PFO, (b) PSO, and (c) Weber-Morris model

Table 1. Original equations and parameters of kinetics models for MB adsorption



PFO model: $ln\ (q_e - q_t) = ln\ q_e - K_1 t$			
$K_1 = 0.1297$ min^{-1}	$R^2 = 0.9433$	q_e, cal. = 8.211 mg/g	Δq_e (%) = 40.76 %
PSO model: $\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{t}{q_e}$			
$K_2 = 0.022$ g/mg. min	$R^2 = 0.9964$	q_e, cal. = 5.626 mg/g	Δq_e (%) = 13.54 %
Weber – Morris model: $q_t = K_{id}\, t^{0.5} + C$			
$K_{id} = 1.467$ mg/g.min$^{0.5}$	$R^2 = 0.9881$	$C = 0.633$	

Adsorption Isotherms

In this regard, the adsorption mechanism was further studied by fitting equilibrium data with Langmuir, Freundlich and Temkin isotherm models at different initial MB concentrations (10–80 mg/L) along with constant temperature (30°C).

Langmuir's model assuming that the surface is uniform and the adsorption of a monolayer occurs on a surface [18], while Freundlich describes adsorption on heterogeneous surfaces [19]. In the case of an adsorbate, which has also been considered in the design of this model, the Temkin model takes into modernised account [20].

Langmuir isotherm characteristics are represented by an equilibrium dimensionless constant (R_L) that allows assessing adsorption favorability and is determined from [18]. It describes four types of isotherms:

$$R_L = \frac{1}{1 + C_0 K_L} \tag{3}$$

$0<R_L<1$, $R_L= 0$, $R_L=1$, and $R_L>1$ correspond to favorable isotherm, non-reversible isotherm, linear isotherm, & unfavorable isotherm respectively.

For the models shown in Figure 3 and corresponding data given in Table 2, Freundlich isotherm model appears to fit best ($R^2 = 0.9919$), suggesting the adsorption happens onto heterogeneous surfaces. According to Langmuir model, it occurred favorably for adsorption with quantification of R_L values from 0.018–0.129; The adsorptive capacity was found to be a maximum of 17.01 mg/g.

Furthermore, an increased Freundlich capacity constant (K_F = 6.525) suggested that MB had higher sorption capacity on PF and also 1/n<1 indicates that the sorption is a chemical process and that the more PF heterogeneous surface [21]. The dye sorption process is exothermic when the positive heat of adsorption (Temkin constant) B>0.

Based on correlation coefficient values, the Freundlich model describes in better way those experimental equilibrium adsorption data of MB compared to other models which suggests non-uniform patches in the heterogeneous surface of material consistent with literature [21].

Table 2 Equations of MB adsorption isotherm models & their parameters

Langmuir model: $\frac{C_e}{q_e} = \frac{1}{q_m K_L} + \frac{C_e}{q_m}$			
$R^2 = 0.9775$	$q_m = 17.01$ mg/g	$K_L = 0.674$	$R_L = 0.018$ to 0.129
Freundlich model: $\log q_e = \log K_F + \frac{1}{n} \log C_e$			
$R^2 = 0.9919$	$1/n = 0.322$	$K_F = 6.525$	
Temkin model: $q_e = B \ln A + B \ln C_e$			
$R^2 = 0.9079$	$A = 37.159$	$B = 2.265$	

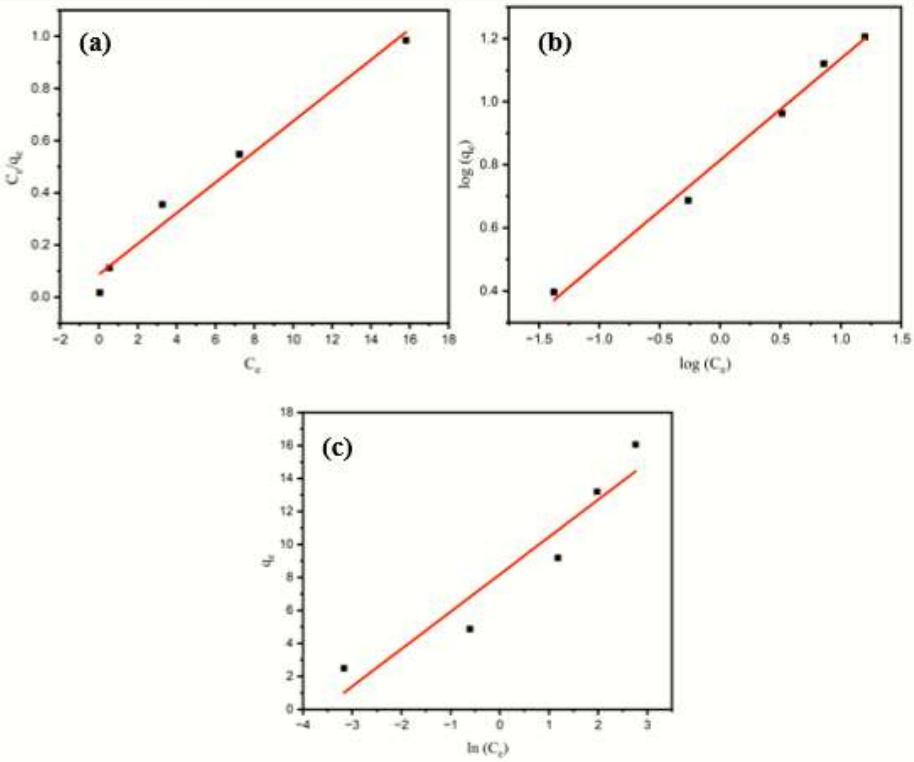
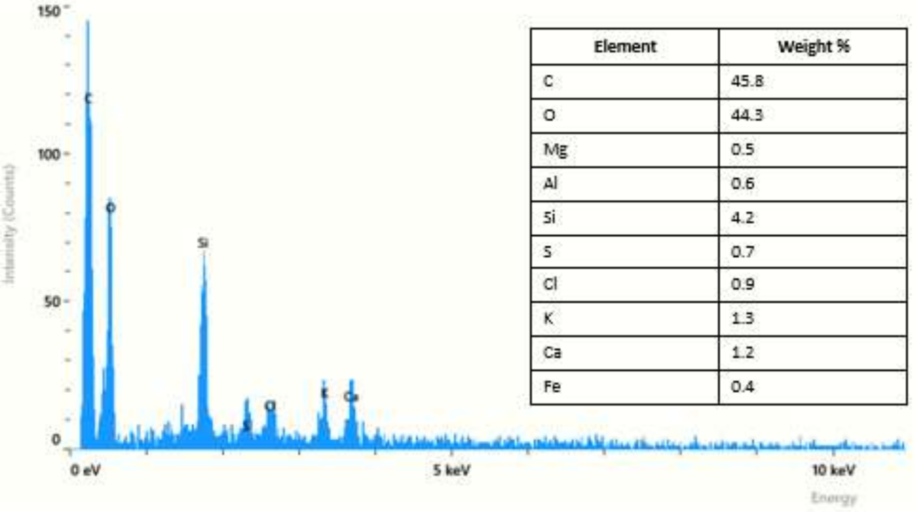


Figure 3 Models of adsorption isotherms: (a) Langmuir; (b) Freundlich; and (c) Temkin

Adsorbent Characterization

EDS Analysis

The EDS analysis (Figure 4) confirmed that carbon and oxygen dominants PF which is why it has a lignocellulosic nature. Such elements are responsible for the functional groups promoting adsorption [22], [23], [24].



Element	Weight %
C	45.8
O	44.3
Mg	0.5
Al	0.6
Si	4.2
S	0.7
Cl	0.9
K	1.3
Ca	1.2
Fe	0.4

Figure 4 EDS analysis

FTIR Analysis

As displayed in Figure 5, the FTIR data showed functional groups of C–O, C=C, O–H, and Si-O were identified; peaks shifting to new positions indicated the interaction between the adsorbent surface and MB molecules upon adsorption which are mainly attributed to hydrogen bonding and electrostatic interactions [21].

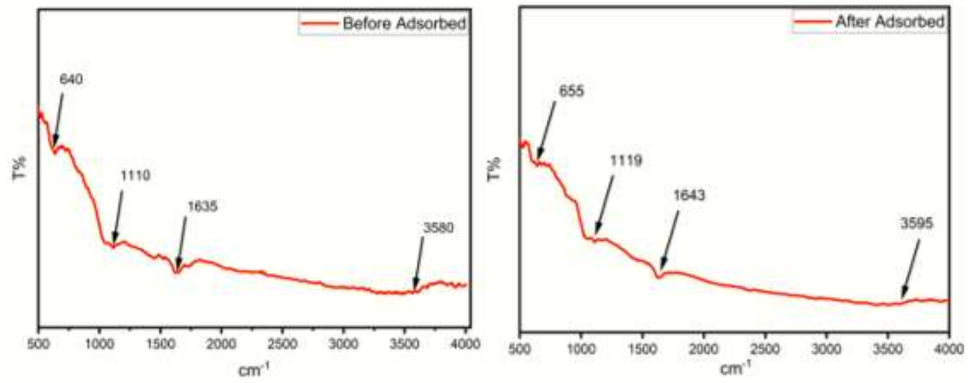


Figure 5 FTIR analysis of PF

SEM Analysis

A PF has an irregular particle morphology and high porosity, as seen from the SEM images of Figure 6 which are essential for imparting a high surface area available for adsorption. This structure enables reactant dye molecules uptake [25].

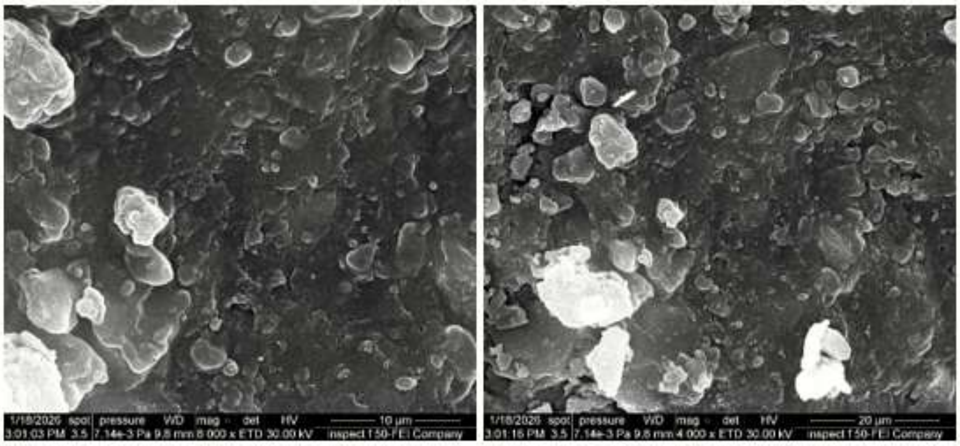


Figure 6 SEM analysis

4. Conclusion

This investigation shows that palm frond becomes an affordable and sustainable adsorbent useful in eliminating methylene blue out of wastewater. Adsorption was studied using different MB concentrations, adsorbent dosage, pH, and time.

As a result, 97.28% removal efficacy was achieved under optimal circumstances (adsorbent dosages of 4 g/L, 60 minutes contact time, pH 12, and 20 mg/L of MB concentration). Kinetic analysis showed pseudo-second-order kinetics, indicating a chemisorption process. The better fit was given by the Freundlich isotherm indicating that heterogeneous adsorption had occurred.

Characterization studies have confirmed PF has an adequate design and practical groups for adsorption. In summary palm fronds are a renewable, low-cost, and locally-available material to be used in the wastewater treatment processes.

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