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Adsorption of Methylene Blue and Reactive Black 5 by Activated Carbon Derived from Tamarind Seeds

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ABSTRACT: One of the most environmentally friendly methods to treat wastewater, especially synthetic dyes, is the production of activated carbon from agricultural waste. Tamarind seeds were transformed from negative-value waste into activated carbon in order to study the removal of synthetic dyes. The particular agro waste was soaked in $ZnCl_2$ for chemical activation to increase its surface area and enhance its porosity. Physical activation of tamarind seeds was done by the carbonization process by burning at a temperature of 300 °C for 1 hour and cooling for 24 hours before washing with HCL to activate a pore surface for the tamarind seeds' carbon. The effects of parameters related to the adsorption of the dyes by tamarind seed activated carbon, such as contact time, initial concentration, absorbance dosage, and pH, were studied. The experimental data found that adsorption on both synthetic dyes exhibited a Langmuir isotherm in which the correlation value, R^2, was 0.9227 (methylene blue) and 0.6117 (Reactive black 5). Meanwhile, the rate of adsorption for methylene blue (MB) and Reactive black 5 (RB5) by tamarind seed activated carbon was found to be well fitted in a pseudo-second-order model. More research is needed to meet the standard effluent of dyeing wastewater from the industrial sector.

KEYWORDS: Activated carbon; adsorption; methylene blue; reactive black 5; tamarind seeds

1. Introduction

In vision 2020, Malaysia needs rapid development in order to move towards becoming a developed nation. One of the factors in achieving the vision is technology developed in the industries.Such developed technology is in the textile industry and dyeing processes. Although dyes are considered pollutants of some water resources, they are compounds that make the world more beautiful through colored products [1]. However, that rapid development, especially in the dyeing industries, would also have a negative impact on the environment. This is due to the current situation, where water pollution has become a major phenomenon. The problem might arise because of the removal of unwanted substances from the water system. For example, textile dye wastewater, which is the effluent from the textile dyeing process, is usually untreated and produced in a large quantity. The dyeing wastewater typically contains high concentrations of chemicals and colors.The improper treatment and bad management of textile wastewater would cause environmental problems [2]. Thus, a very effective method is needed in order to treat the wastewater from the dyeing textile industries.

Wastewater decolorization in various industries has become a major concern for the treatment plants that treat the wastewater. Wastewater treatment usually involves a combination of physical, chemical, and biological unit processes. Conventional wastewater treatment plants (WWTPs) are normally efficient, but additional water treatment is required to remove a wide range of pollutants [3, 4]. A series of single-unit processes, with the effluent of one process becoming the influent for the next process, are usually processes that were involved in conventional treatment. Many conventional wastewater treatment techniques are used to remove dye from wastewater. Electrochemical coagulation, reverse osmosis, nano filtration, and adsorption using activated materials are examples of conventional wastewater treatment methods [5, 6].

Tamarind, or its scientific name, *Tamarindus indica* Linn, is useful as an anthelmintic, antidiarrheal, and anti-emetic. Although it is known that tamarind seeds contain phenolic substances, the single components of the seeds have not been identified and quantified. Extraction of tamarind pericarp and seeds using acetone, methanol, and acetic acid gave only procyanidin oligomers in a much higher yield and variety. Aside from being a fruit component, tamarind seeds are commercially and nutritionally valuable, as well as having the ability to improve epidermal wound healing [7, 8]. Tamarind seeds can be considered as a potential source material for the preparation of activated carbon. Previous studies have explored the adsorption process in which tamarind seed powder is used as an adsorbent to remove crystal violet dye by using different variables, and pH was found to be the most effective variable. Different adsorbent properties such as surface area, porosity, and physical strength would have different effects on adsorption capacity. Available adsorbent would certainly make an adsorption process a viable alternative for the treatment of wastewater containing pollutants. In order to achieve the maximum removal of a type of pollutant, the selection of a suitable adsorbent is important depending on the adsorbent and adsorbate characteristics [9, 10].

Adsorption has been an effective separation process for non-biodegradable pollutants. It can be proved through research done by many researchers in approximately 25 years that they have studied the feasibility of low-cost adsorbents prepared from natural materials, industrial materials, agricultural waste, and bioadsorbents and found an innovative approach in this area. The process of adsorption has a sludge-free, clean operation and is able to remove dye completely even from diluted solutions. The adsorption process is one of the physicochemical methods to treat wastewater by using activated carbon. The adsorption process is effective in the removal of hazardous materials such as dyes and this method has gained considerable attention in recent studies [11, 12]. For example, adsorption of MB from the surface of sheep wool and cotton fibers was done under the optimal conditions of temperature, concentration of dyes, pH, retention time, and amount of adsorbent. Treatment of dye wastewater by using the adsorption method involves only phase change of pollutants and imposes further problems such as sludge disposal. The expensive cost of the adsorbent for the adsorption process also necessitates its regeneration for wastewater treatment [13].

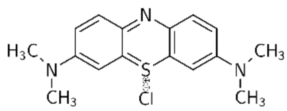
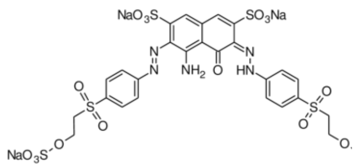
Commercial activated carbon is preferred by most textile industries to treat dye wastewater. This might be due to the ability of activated carbon to recover both inorganic and organic compounds from gas and liquid streams. Activated carbon is high in adsorption capability because it has a high internal surface area and more porosity formed during the carbonization process in order to obtain char and carbonaceous materials. Adsorption is greatly affected by the physical factors of the pores, including the size, shape, distribution, and specific surface area. The methods used for activated carbon synthesis are physical activation and chemical activation. In physical activation, the precursor is carbonized in an inert atmosphere and then activated in a stream of carbon dioxide or steam, whereas for chemical activation, the precursor is impregnated with a dehydrating agent. Usually used are zinc chloride or inorganic acids, prior to carbonization in an inert environment [2]. The objective of the study was to investigate the ability of tamarind seed activated carbon for the adsorption of MB and RB5.

2. Materials and Methods

2.1. Materials.

The activated carbon employed in this adsorption investigation was derived from tamarind seeds. The tamarind was washed in order to extract the seeds and then stored in a container. Meanwhile, this study employs two types of synthetic dyes: MB and RB5. Both dyes are from the laboratory stock supplied by Sigma Aldrich (USA). Table 1 summarizes the physical and chemical properties of MB and RB5.

Table 1. Physical and chemical characteristics of MB and RB5.

Properties	Physical-chemical	
Name	Methylene Blue (MB)	Reactive Black 5 (RB5)
Structure		
Molecular formula	$C_{16}H_{18}ClN_3S$	$C_{26}H_{21}N_5Na_4O_{19}S_6$
Appearance	Dark green crystal	Black powder
Molecular weight	319.85	991.81
Melting point	100-110 °C	300°C
Solubility	43.6 g/l	200 g/l
Absorption, λ_{max}	664 nm	611 nm

2.2. Preparation of the stock solution.

A stock solution of 100 ppm MB has been prepared. A solution of 0.1 g MB in 1 L distilled water was prepared, followed by dilution in a 500 ml volumetric flask. A calibration and standard curve were created for the specific MB and RB5 concentration. RB5 is a dye based on the vinyl sulphone reactive group that has a negative charge in aqueous solution. A stock solution of 200 ppm was made by dissolving 0.2 g of dye in 1 l distilled water The MB and RB5 dyes were analyzed spectrophotometrically by monitoring the absorbance between 600 and 700 nm using a UV-VIS spectrophotometer.

2.3. Activated Carbon Preparation.

The raw tamarind seeds (TS) were cleaned and rinsed with tap water and distilled water. The tamarind seeds were then dried overnight in an oven set to 110°C. For 24 hours, dried tamarind seeds were soaked in $ZnCl_2$ in a 1:1 ratio with their weight. $ZnCl_2$ was chosen as the reagent to soak with tamarind seeds due to its capacity to chemically activate the seed's surface area and porosity. Following that, the activated tamarind seeds were carbonized in a horizontal furnace (N_2 at 300°C for 1 hour, N_2 flow rate 6.00/min). When tamarind seeds were carbonized, they undergo physical activation to increase their surface area, porosity, and strength. After that, the activated carbon was rinsed with a 10% HCl solution to eliminate any remaining contaminants. Tamarind seeds that have been activated are rinsed with distilled water until the pH of the washed water reaches neutral (pH 7). After approximately 3 hours of drying at 110°C, activated carbon generated from tamarind seeds was grained until fine and stored at room temperature [15].

2.4. Activated carbon characterization.

The functional groups on the surface of the raw tamarind seeds and tamarind seed activated carbon were analyzed using Fourier Transform Infrared Spectrometry (FTIR) (Spectrum One, Perkin Elmer, USA). The surface morphology of the raw tamarind seeds, TS-activated carbon, and TS-activated carbon after the adsorption process were investigated using a Field Emission Scanning Electron Microscope (FESEM, JEOL 6335F-SEM, Japan), and elementary analyses were performed simultaneously using an EDX spectrometer.

2.5. Adsorption test.

Adsorption tests are carried out based on the study objectives. In order to optimize the contact time, initial dye concentrations, initial adsorbent dosage, and pH of the two types of dye (MB and RB5) into tamarind seed activated carbon, each parameter was varied accordingly after running pre-experiment to the related objectives. Batch experiments were performed in 100 ml conical flasks by introducing 50 ml of dye solution of various concentrations, pH, contact time, and initial dosage of activated carbon (Table 2).

Table 2. Summarizes for all experiments of batch adsorption.

Parameters	Initial pH	Dosage (g)		Time (min)		Initial Concentration (mg/l)	
		MB	RB5	MB	RB5	MB	RB5
Effect of Initial pH	1 - 14	0.35	4	50	140	17.5	70
Effect of Dosage	7	0.04-0.6	2-6	50	140	17.5	70
Effect of Contact Time	7	0.35	4	9-90	17-264	17.5	70
Effect of Initial Concentration	7	0.35	4	50	140	2-33	8-132

2.6. Adsorption Isotherm and kinetics.

In order to determine the nature of adsorption between the activated carbon with methylene blue and the activated carbon with RB5 dye was determined. This basic technique indicates the distribution of both synthetic dyes in the equilibrium phase. The adsorption isotherm was analysed using the Langmuir isotherm model and the Freundlich isotherm model. The Langmuir and Freundlich models are the most commonly used models to evaluate the experimentally acquired adsorption isotherms. The Langmuir equation is applicable to homogenous sorption. Meanwhile, the Freundlich equation is applicable to heterogeneous sorption [16]. Equations for the respective isotherm models are given below.

Langmuir equation $$\frac{C_e}{q_e} = \frac{1}{K_L q_m} + \frac{C_e}{q_m} \quad (1)$$

Freundlich equation $$In q_e = In K_F + (\frac{1}{n}) In C_e \quad (2)$$

The rate of adsorbate uptake during the adsorption is evaluated by the reaction kinetics. The dye sorption kinetics was investigated by examining the influence of contact time on the dye removal for 90 min (MB) and 140 min (RB5). The conditions of the kinetic procedure were identical to those of the equilibrium procedure. The aqueous samples were taken at intervals of time and the concentrations of residual dyes were measured. The kinetics data for MB and RB5 adsorption were treated with the following pseudo-first-order and pseudo-second-order models:

Pseudo-first-order equation $$\log(q_e - q_t) = \log q_e - k_1 t/2.303 \quad (3)$$

Pseudo-second-order equation $$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{t}{q_e} \quad (4)$$

where T is contact time with adsorbent (min), qm is maximum adsorption capacity (mg/g), q_e is number of dyes absorbed at equilibrium time (min), K_L is Langmuir rate constant, q_t is adsorbed amount at time t min (mg/g), k_1 is pseudo-first-order rate constant, and k_2 is pseudo-second-order rate constant.

3 Results and Discussion

3.1. Characteracterization Study.

The purpose of FESEM analysis was to study the surface morphology of the tamarind seeds in their raw state, soaked with ZnCl2, activated carbon, and activated carbon after treatment. The images from the FESEM analysis using 500X magnification are presented in Figure 1. Through observation, the raw sample of tamarind seeds is non-porous. This might be because no activation process occurred in the raw sample of tamarind seeds. This can be attributed to the chemical activation using $ZnCl_2$, which plays a main role in developing the formation of pores through the decomposition of water and other organic compounds. Figure 1B shows an image of carbonized tamarind seeds, which are now called tamarind seeds activated carbon. From the observation, the number of pores increased, and the surface area of the adsorbent became larger. However, after treatment, the pore size is reduced, and the number and surface area become smaller and harder to see. Figure 1C shows the activated carbon of tamarind seeds that have been used in the batch adsorption study for MB dye.

On the other hand, FTIR spectroscopy analysis was done to determine the functional group that was involved in MB and RB5 dye adsorption into tamarind seed activated carbon. Each state of tamarind seeds was presented in Fig. 2. The FTIR showed that the tamarind seeds' activated carbon has different adsorption peaks. A broad band was observed at Figure 2A (3477.42 cm^{-1}), followed by Figure 2B (3388.49 cm^{-1}) and Figure 2C (3410.26 cm^{-1}). The broad band region occurred because of the stretching frequency of the hydroxyl (OH) group. Figures 2C (1629.19 cm^{-1}), 2B (1625.80 cm^{-1}) and 2A (1625.37 cm^{-1}). There is a decrease in transmission following the adsorption of synthetic dyes into activated carbon from tamarind seeds.When the functional group is stretched, increasing and decreasing transmission can be observed.The functional group found in the sample might be a potential active site for the adsorption process to take place.

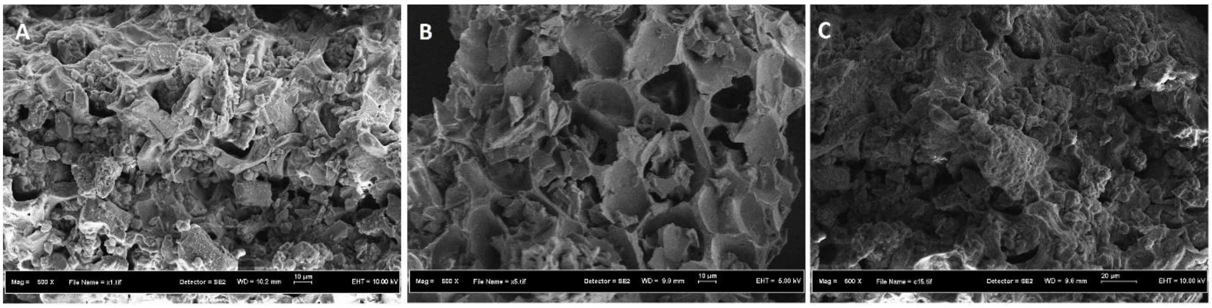


Figure 1. SEM of tamarind seed: raw (A), activated carbon (B), and after treatment (C).

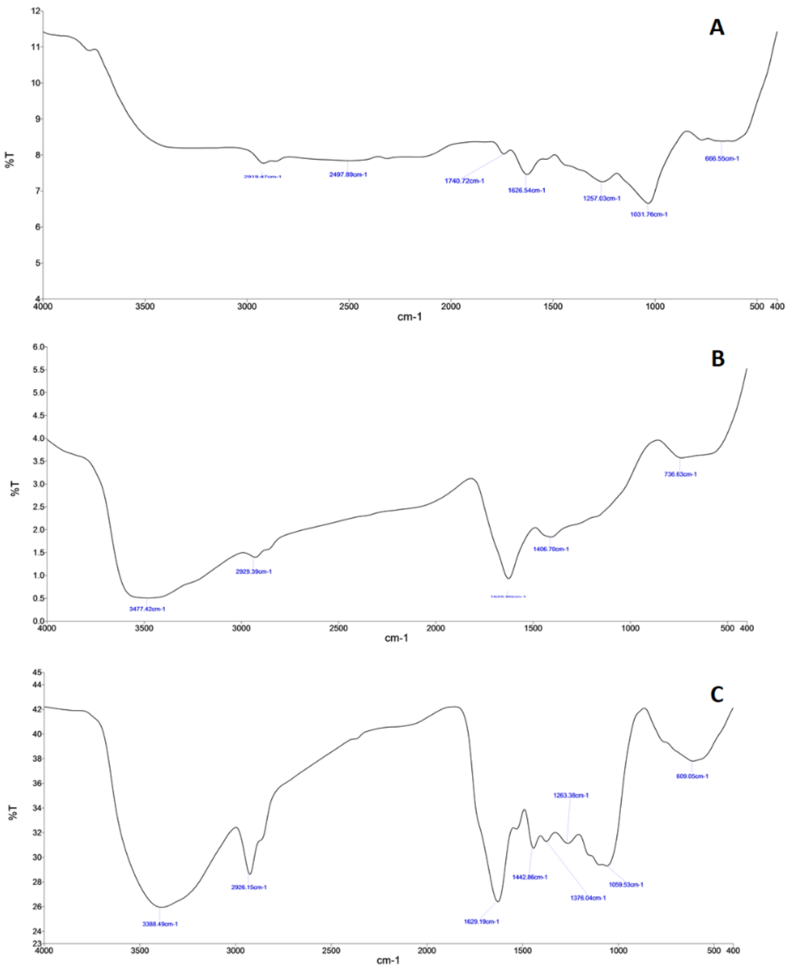


Figure 2. FTIR of tamarind seed: raw (A), activated carbon (B), and after treatment (C).

3.2. Effect of pH.

The adsorption of MB dye by tamarind seeds was significantly greater in acidic than in alkaline conditions (Fig. 3A). At pH 4, the highest percentage of MB removal was observed. A previous study discovered that at lower pH values, the removal of methylene blue was 40%, and that at higher pH values, the removal trend increased. Adsorption of the MB dye increased as the pH was increased from 2 to 7. MB is a cationic dye that exists in aqueous solution as positively charged ions [14]. The experimental data indicated that the best conditions for adsorption of RB5 dye into activated carbon derived from tamarind seeds are pH 11, which was an alkaline condition. The percentage of dye RB5 removal increases as the dye solution becomes more alkaline, until it reaches pH 14, at which point the percentage of dye RB5 removal decreases. Meanwhile, dye adsorption was reduced when the solution is acidic compared to when the solution is alkaline. A previous study demonstrated that the maximum adsorption efficiency of RB5 occurred after 120 minutes at pH 7. The adsorption of the RB5 onto an adsorbent generally varies with pH, as the radius of the hydrolyzed cation and the charge on the adsorbent surface are affected by pH [17]. The concept is as follows: When the pH solution is increased, the surface of the catalyst becomes negatively charged by adsorbed hydroxyl ions, and when the pH solution is decreased, the surface becomes positively charged by adsorbed hydrogen ions. Due to the change in electrostatic forces between surface catalysts and dye molecules, both acidic and basic media have an inverse effect on adsorption efficiency [18]. On the contrary, previous study discovered that as the pH increased, the efficiency of dye removal decreased, and that an acidic pH was more favorable for RB5 dye adsorption in their experiments [19].

3.3. Effect of initial concentration.

The result showed that the percentage of removal decreased gradually as the initial concentration of dyes increased (Fig. 3B). The percentage removal of dye is highly dependent on the initial amount of dye concentration. Similar results from previous studies also showed that the adsorption percentage decreases and the extent of adsorption increases with increasing initial dye concentration. The increase in initial dye concentration will cause an increase in the loading capacity of the adsorbent. This might be due to the high driving force for mass transfer at a high initial dye concentration [20,21]. The removal efficiency was decreased with an increase in initial concentrations, although the amount of total methylene blue accumulation increased. The total accumulation of methylene blue increased with increasing initial concentration, which was probably due to more contact of adsorbent sites with methylene blue. The dye initial concentration increased from 10 to 100 mg/l and the dye removal efficiency declined from 100% to 77.97%, respectively [21,22].

3.4. Effect of adsorbent dosage.

The result showed that the increase in initial dosage was linear with the percentage of dye removal (Fig. 3C). The higher the dosage of adsorbent, the greater the rate of adsorption. Previous studies indicated that dye removal efficiency increased with the adsorbent dosage. This is due to the availability of more binding sites as the dose of biosorbent increases. Study of the effect of adsorbent dosage gives an idea of the effectiveness of an adsorbent and the ability of dye to be adsorbed with a minimum dosage. They indicate that dye removal efficiency increased with the adsorbent dosage. A study has observed that, apparently, the removal percentage of MB dye increases as the adsorbent amount increases and then becomes constant ([14, 20]. The percentage of MB uptake increases with increasing adsorbent dosage at the same initial MB concentration. Adsorption of RB5 dye by activated carbon found that an increase in biomass dosage was able to increase the adsorption rate [19]. By increasing the catalyst amount, the adsorption efficiency increases, but the adsorption density and the amount adsorbed per unit mass decrease. It is easily understood that the number of available adsorption sites increases by increasing the adsorbent amount, but the drop in adsorption capacity is basically due to the sites remaining unsaturated during the adsorption process. If the active sites are available, the pollutant left in the system will be continuously adsorbed. The removal of RB5 dye increased as the weight of the initial dosage became larger. Therefore, the adsorption efficiency of RB5 increased with increasing adsorbent dosage [17].

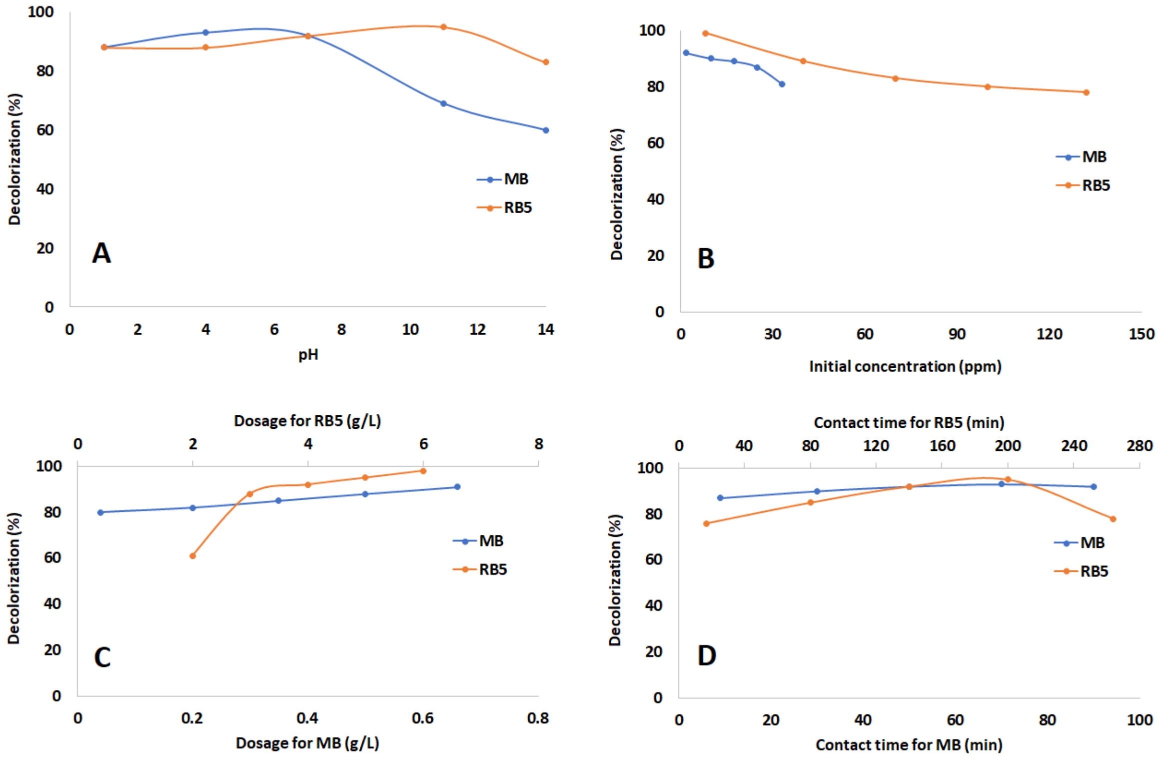


Figure 3. Effect of pH (A), Initial concentration (B), Dosage (C), and Contact time (D) on degradation of MB and RB5.

3.5. Effect of contact time.

The result showed that the percentage of removal are increased when contact time increased (Fig. 3D). However, after minutes of 70 the percentage of removal start to decrease slowly. Previous studies indicated that the sorption of MB increased with increasing contact time initially and became almost constant after 114 min of contact in their study. This might be caused by the saturation point was achieved and no more adsorption process occurred. The rate of color removal increased depending on the contact time. the result from their experiments showed that for the first 60 minutes, the percentage removal for MB dye by the adsorbent was rapid and thereafter it proceeds at a slower rate and finally attains saturation at different contact time for different initial concentration of the dye. This might be caused by after a certain period, only a very low increase in the dye uptake was observed because there are few active sites on the surface of sorbent [14, 20]. From their result, we could indicate that the state of dynamic equilibrium in which the amount of dye adsorbed onto the adsorbent is in equilibrium with that of dye present in solution. However, at minutes 200, the percentage of removal dropped down from 95% to 78%. This indicate that, after 200 minutes, the adsorption process achieved saturation point and no adsorption occurred. Previous studies found that the same result and equilibrium was attained at 105 minutes for RB5 dye and the adsorbed amount of RB5 increases with the increase of contact time [18, 19].

3.6. Adsorption isotherm.

An isothermal study is a basic technique for determining the nature of adsorption between the activated carbon and dye solution. It indicated the distribution of dyes in the equilibrium phase. Table 3 shows the adsorption isotherm parameters of dye adsorption. The Langmuir model equation showed it's applicable to homogenous sorption. In this experiment, both dyes were found to be at their best fit in the Langmuir model. The R^2 of MB (0.9097) and RB5 (0.6117) was higher if compared with the Freundlich model of isotherm. This indicates that the sorption process is homogenous.

Table 3. The isotherm model in the adsorption of MB and RB5.

Adsorption isotherm	MB	RB5
Langmuir model		
q_m (mg/g)	1.42	1.62
K_L (L/mg)	-3.22	-1.54
R^2	0.9227	0.6117
Freundlich model		
K_F (m/g) (L/mg)$^{1/n}$	0.86	0.67
n	1.42	1.62
R^2	-0.201	0.3896

The Langmuir isotherm provided the best fit for MB adsorption onto the prepared activated carbon. Previous studies showed that methylene blue adsorption on activated carbon as well as water hyacinth ash from aqueous solutions and found that the Langmuir isotherm model has been found to be more accurate compared to the Freundlich isotherm [12,20]. The experimental results are well represented by the Langmuir model, with a higher value of the correlation coefficient. Another similar result was found for the adsorption of methylene blue, which was able to adsorb strongly onto the surface of the sunflower seed husks-based activated carbon and the equilibrium data were best described by the Langmuir isotherm model, with maximum monolayer adsorption capacity. The result is compared with previous studies and most of them agree that the adsorption of MB is best fitted at Langmuir compared with the Freundlich model. The result showed a linear curve in the Langmuir isotherm model with a correlation factor of R^2 = 0.6117. This suggests that tamarind seed activated carbon has a homogenous surface where each site of tamarind seed activated carbon can accommodate one molecule of dye. The Langmuir model fitted the experimental data better than the Freundlich model, indicating that the adsorption tends to monolayer adsorption [18, 20]. Yousefi et al. showed that RB5 adsorption by modified wheat straw found that the adsorption isotherm most favors the Langmuir isotherm compared with the Freundlich isotherm model [22]. Studies conducted by Samadi et al. showed that the results illustrated among isotherm data were fitted well with the Freundlich model with a correlation coefficient of 0.91. This can be due to different sites with several adsorption energies being involved [17].

3.7. Adsorption kinetic.

Adsorption of MB and RB5 by activated carbon prepared from tamarind seeds is in the pseudo-second-order of kinetic isotherm study (Table 4). Both R^2 values showed that the adsorption was best fit compared to the pseudo-first-order graph. Adsorption of RB5 by synthesized titanium dioxide nanoparticles showed good agreement with a pseudo-second-order kinetic model [18]. The rate of reaction of the RB5 adsorption by palm kernel shell activated carbon was such that the analysis of contact time experiments suggests that the pseudo second-order kinetics model described the dynamic behavior of the adsorption well. Another study discovered that adsorption of RB5 dye by modified wheat straw is best with pseudo-second-order [22].The pseudo second-order equation was more appropriate in his study, which involved the adsorption of RB5 by using chitin as an adsorbent. The results obtained revealed that the adsorption process followed the second-order kinetic model with a correlation coefficient value of 0.91 [17].

Table 4. The kinetic model in the adsorption of MB and RB5.

Kinetic model	**MB**	**RB5**
Pseudo-first-order		
q_e (mg/g)	0.33	0.11
K_1	0.005	0.005
R^2	0.2696	0.8642
Pseudo-second-order		
q_e (mg/g)	0.33	0.11
K_2	4.29	66.84
R^2	0.9097	0.9938

4. Conclusion

Both synthetic dyes were effectively removed from aqueous media by tamarind seeds' activated carbon. The adsorption time for MB blue is 30 minutes, with 95% removal of the dye. Meanwhile, for RB5, the adsorption time was 140 minutes with 99% removal of dye. The isotherm data showed a better correlation with the Langmuir isotherm model than the Freundlich isotherm model. The best R^2 value obtained from the kinetic model for MB was 0.9097 in pseudo-second-order, which is higher than the pseudo-first-order kinetic model. Study of the kinetic model on RB5 also showed that the R^2 value obtained in the pseudo-second-order model is 0.9938, which is higher than the pseudo-first-order model. This indicated that the adsorption process obeyed the pseudo-second-order model for the entire adsorption period. The removal of MB and RB5 was found to be favourable, and tamarind seed activated carbon can be an alternative commercially viable absorbent.

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Conflicts of Interest

The authors declare no conflict of interest.

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