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Removal of Methylene Blue Using Durian Peel: A Column Experiment

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ABSTRACT

The increasing demand for sustainable, low-cost alternatives to activated carbon has spurred the use of agricultural waste, such as durian peel (DP), in wastewater treatment. This study evaluates the performance of DP, a lignocellulosic material rich in cellulose, hemicellulose, and lignin, for the removal of Methylene Blue (MB) from aqueous solutions using a fixed-bed column. Experiments were conducted in a column of 1.5 cm diameter at bed depths of 2, 3, and 4 cm and flow rates of 3, 4, and 5 mL/min, with an initial MB concentration of 50 mg/L. The treated volumes increased with bed depth, achieving 294, 594, and 1044 mL for 2, 3, and 4 cm, respectively. At a constant bed depth of 3 cm, lower flow rates improved adsorption efficiency, with treated volumes of 940, 594, and 230 mL at flow rates of 3, 4, and 5 mL/min, respectively. Breakthrough occurred within 2–3 h, indicating near saturation of the adsorption zone. The experimental breakthrough curves exhibited typical adsorption profiles and were well described by the Thomas and Yoon–Nelson models (R^2 = 0.95-1.00), with predicted breakthrough times closely matching experimental values. The findings demonstrate that DP is an effective and environmentally friendly adsorbent for dye removal from wastewater.

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INTRODUCTION

Water is essential to our daily lives. According to the U.S. Geological Survey, about 96.5% of Earth's water is saltwater and is not suitable for human consumption

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without further treatment. About 3.5% of the total water is fresh water. Only 0.8% of freshwater is available to humans, agriculture, and industry, and it is mainly stored in polar ice and underground sources. Due to the increasing global population and industrial activity, the demand for water is also rising. The fresh water sources from rivers, lakes, and aquifers were meant to supply water for agriculture, industry, and households; however, these supplies have increased greatly in the last decade, from 600 km3 to more than 4000 km3 per annum (Dawood, 2013). By 2025, it was estimated that about 60% of the world's population would live in places experiencing moderate-to-extreme water-resource-based vulnerability (Kulshreshtha, 1998).

With urbanisation and industrialisation over the years, water is becoming polluted. Dyes are an important class of pollutants that, once they enter water bodies, can harm ecosystems. Several industries use dyes in their production, including textiles, dyestuffs, paper, printing, plastics, food, and cosmetics. All these dyes will be left as industrial waste and directly discharged into the water body. In Malaysia, the same problem is currently being faced and has been a major problem for environmental management (Hameed & Hakimi, 2008). Dyes can be classified into cationic, anionic and non-ionic dyes. Out of those classes of dyes, the removal of anionic dyes is known to be the most challenging task, as they are water-soluble and produce very bright colours in water with acidic properties. It has been estimated that the total dye consumption in the textile industry worldwide exceeds 10,000 tons per year, and about 10-15% of these dyes are released as effluents during dyeing processes (Gupta et al., 2013).

Various methods have been developed to remove dye wastewater, such as adsorption, anaerobic treatment, coagulation, electrocoagulation, flotation, filtration, ion exchange, membrane separation, and advanced oxidation (Robinson et al., 2001). However, there are many limitations to those treatments, including low efficiency, high operational costs, corrosion, recontamination, incomplete removal of coloured effluent (for dyes), and more. Additionally, adsorption is a separation method for removing dye-bearing wastewater, as reported by Kumar et al. Furthermore, activated carbon is a widely used adsorbent. It is not an economical way to use activated carbon, especially for treating dye wastewater (Wang et al., 2005); hence, many researchers have actively explored alternative, cheaper materials in recent years.

Various low-cost adsorbent materials derived from agricultural waste have been studied over the past few decades (Yagub, 2013). There are many advantages to agricultural wastes, such as being cheap and abundantly available, requiring simple technology, having relatively low running costs, and being biodegradable. Durian peels, an agricultural waste or by-product, have been selected as adsorbents in this study. The utilisation of durian peels as an adsorbent has been studied by several researchers, and it has been shown that they are promising sorbents for removing dyes from aqueous solutions (Hameed & Hakimi, 2008).

In this study, methylene blue (MB), a basic dye, is selected as a model compound to evaluate the capacity of durian peels (DP) to remove MB from solutions. MB, the most common dye in its category, is generally used to dye cotton and silk. Even though MB is not highly hazardous, it can still cause harmful effects. Acute exposure to MB can cause increased heart rate, vomiting, shock, Heinz body formation, cyanosis, jaundice, quadriplegia, and tissue necrosis in humans (Vadivelan & Vasanth Kumar, 2005).

This study assesses the feasibility of using durian peels as an adsorbent for removing dyes from wastewater. The study was conducted with specific objectives: to determine the effect of column parameters on MB removal by durian peels and to simulate the experimental data using column kinetic models, such as the Thomas and Yoon-Nelson models.

This study was established to achieve its objectives. Furthermore, it is a promising approach for developing an environmentally sustainable adsorbent to treat wastewater and remove MB. On top of that, this study aims to analyse the effects of parameters such as bed heights (2-4 cm) and flow rates (3-5 mL/min). This study used a column design to investigate breakthrough plots for these parameters. Thomas and Yoon-Nelson mathematical models were applied in this study to analyse the breakthrough curves (BTCs) on the MB adsorption.

The absorbance of dyes depends on dye characteristics, including molecular structure and the type, number, and position of substituents. Agricultural by-products, which are cheaper than activated carbon, are readily available and can be used directly as adsorbents or for partial treatment. The advantages of using agricultural by-products as adsorbents include lower cost, high efficiency, the ability to minimise chemical and biological sludge, and the possibility of dye recovery.

Thus, the importance of this study is also to identify or explore another environmentally friendly and economically beneficial agricultural by-product for use as an adsorbent, given its ease and affordability in treating wastewater. The purpose of this research is to use durian peels to efficiently remove MB. Moreover, conducting research on this will raise public awareness of the environment and strengthen the urge to protect the quality of water and wastewater in the future.

MATERIALS AND METHOD

Preparation of Durian Peel

Durian Peel (DP) was collected from the local market in Shah Alam, Selangor. The biomass was rinsed repeatedly with distilled water to remove dirt and dust, but the colour persisted. The cleaned biomass was first dried in sunlight and then oven-dried overnight at 80°C. The dried biomass was ground with a domestic blender and lastly sieved to obtain a particle size range of 500 to 1000 μm. The prepared biomass was stored in a plastic container and used for further adsorption experiments.

Preparation of Aqueous Dye Solution

The cationic dye Methylene Blue (MB), with a λmax of 665 nm, was selected as the model adsorbate in this study. It has the formula of C16H18ClN3S and a molecular weight of 319.85 g. The analytical grade was obtained from Friendemann Schmidt with 99% purity. A stock solution of dye was prepared by dissolving 50 mg of MB in 500 mL of distilled water.

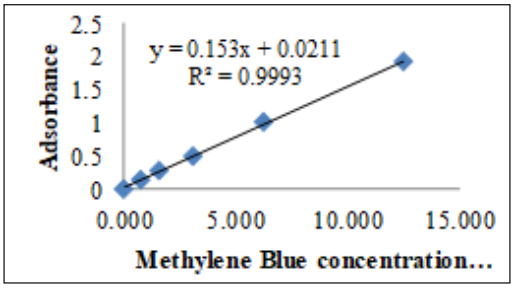

Figure 1. UV Calibration curve plot for MB adsorption

Spectrophotometer

The DR2800 spectrophotometer was used to measure the concentration of the MB dye in solution. It was measured at ʎ max. The maximum adsorption of the dye solution was measured at λmax = 665 nm using a spectrophotometer.

Calibration Curve

Figure 1 shows the calibration curve for the adsorption of MB dye at concentrations ranging from 0.00 to 12.50 ppm, with a regression coefficient of 0.9993.

Column Design Experiment

The adsorption of MB dye on durian peels was carried out in a fixed-bed column design. The experimental arrangement consists of a peristaltic pump, a reservoir containing dye stock solution, a 14 mm-diameter silicon tube delivery line to convey the dye solution from the reservoir to the column, an adsorbent-packed column, and vials serving as the sampling point, as shown in Figure 2. A fixed-bed column study was performed using polypropylene

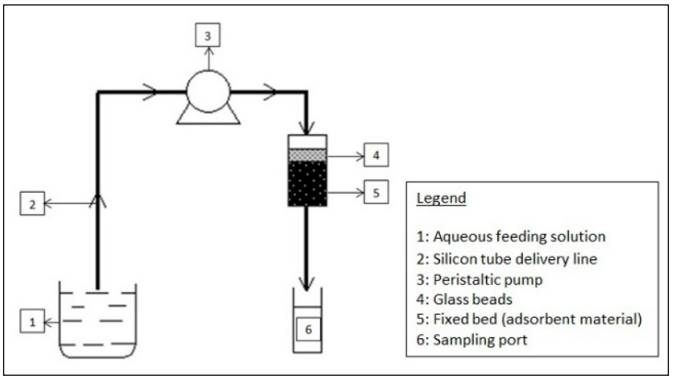

Figure 2. Column system schematic drawing

cylindrical columns with diameters and heights of 1.5 cm and 7.5 cm, respectively. The column was then occupied with a net cloth bed of 1 mm at the bottom and top sections to hold the adsorbent, whilst ensuring a homogeneous distribution of the feed solution. Using a 14-gauge silicon tube with a Cole-Palmer Masterflex peristaltic pump, a 50-ppm dye stock solution was pumped into the down-flow mechanism at the desired flow rate. Samples of column runoff were collected in intervals using vials and were examined for absorbance. The column operation is stopped once the effluent MB concentration exceeds 99.5%, as suggested by Uddin et al. (2009).

Effect Column of Parameters

Two operating parameters were studied. In the bed-depth study, different volumes of adsorbent were packed into the column in each run to provide bed depths of 2, 3, and 4 cm, while the flow rate and MB concentration were constant at 4 mL/min and 50 ppm, respectively. As for the flow-rate study, the analogous fixed-bed experiment was repeated at flow rates of 3, 4, and 5 mL/min, with the bed depth and MB concentration held constant at 3 cm and 50 ppm, respectively.

Thomas Model

The Thomas model was used to characterise the performance of the fixed bed for MB removal and to predict the concentration-time profile, or breakthrough curve, of the effluent. The input data were nonlinear plots of effluent concentration versus sampling time from experimental findings for both bed-depth and flow-rate studies. The output data of this model were the Thomas rate constant (kTh), equilibrium adsorbate uptake (q0) and regression coefficient (R2). Only R2 was discussed in this column study.

Yoon-Nelson Model

A simple theoretical model developed by Yoon–Nelson was applied to investigate the breakthrough behaviour of MB on durian peel. The input data were nonlinear plots of effluent concentration versus sampling time from experimental findings for both bed-depth and flow-rate studies. The output data for this model were the Yoon-Nelson rate constant (kYN), the time needed for 50% adsorbate breakthrough (τ) and the regression coefficient (R2). Only τ and R2 were discussed further in this column study. The τ values from the model represent the time required for 50% adsorbate breakthrough and are used to compare with the time required for 50% adsorbate breakthrough from experimental findings. While the R2 value was the regression coefficient.

RESULTS AND DISCUSSIONS

Effect of Bed Depth on Breakthrough Curve

To study the effect of bed depth on the breakthrough curve, a column experiment was carried out at bed depths of 2, 3, and 4 cm, whilst the MB concentration and the flow rate were held constant at 50 ppm and 4 mL/min, respectively.

As shown in Figure 3, the adsorption bed was exhausted within 1 to 3 hours after breakthrough, indicating that the adsorption zone was almost saturated at the time of exhaustion. These types of breakthrough curves are highly desirable. The breakthrough times, corresponding to C/C0 = 0.200, were 1.25, 13.75, and 26.25 minutes for bed depths of 2, 3, and 4 cm, respectively. The exhausted times, corresponding to C/C0 = 0.995, as suggested by Uddin et al. (2009), were 58.75, 118.75, and 208.75 minutes, respectively. The corresponding volumes of wastewater treated at the breakthrough point were 6.25, 68.75, and 131.25 mL, respectively, and at the exhaust point were 293.75, 593.75, and 1043.75 mL, respectively.

In Figure 3, the breakthrough curves showed that as the bed depth increases, MB has more time to contact durian peels, resulting in improved MB ion removal performance in the column. Therefore, the solute concentration in the effluent decreased simultaneously with the increment in the bed column results. A wider mass-transfer zone resulted from the decreasing slope of the breakthrough curve as bed depth increased. As a result of the increase in the adsorbent surface area, additional adsorption sites were created, as evidenced by the high uptake observed at the topmost bed depth (Han et al., 2007).

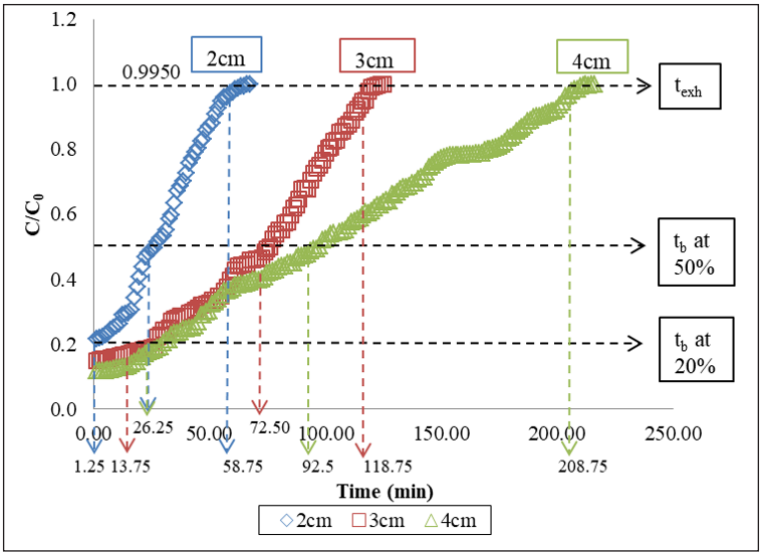

Figure 3. Bed depth effect on breakthrough curve on durian peels mb adsorption at C0 = 50 mg/L and 4 mL/min flow rate

Table 1
Column experiment breakthrough parameters on bed depth

Bed Depth	HRT (min)	T_b (min)	V_b (mL)	t_{exh} (min)	V_{exh} (mL)	% Removal
2 cm	0.8837	1.25	6.25	58.75	293.75	49.05
3 cm	1.3255	13.75	68.75	118.75	593.75	80.93
4 cm	1.7674	26.25	131.25	208.75	1043.75	82.49

As illustrated in Figure 3, a common breakthrough curve was plotted to investigate the effect of bed depth on MB removal. From this parameter study, the column parameters information was gathered and tabulated in Table 1. According to Table 1, the longest breakthrough time was observed at a bed depth of 4 cm (26.25 minutes), compared to 2 cm and 3 cm, which were only 1.25 and 13.75 minutes, respectively, thereby providing more contact time with the adsorbent to adsorb a larger volume of MB. This indicates that the lower bed depth reached breakthrough sooner than the higher bed depth, and that the column exhaustion time increased significantly with bed depth. It was observed that the breakthrough curves became steeper as the bed depth reduced. Although MB adsorption was rapid, diffusion effects were reduced due to insufficient dye residence time in the column at lower bed depths. These results show that higher bed depth yields better MB removal performance than lower bed depth in column mode.

As shown in Table 3, the column reached the exhaustion condition more slowly at a 4 cm bed depth (208.75 minutes) than at 3 cm (118.75 minutes) or 2 cm (58.75 minutes), respectively. In addition, higher bed depth produced a larger volume of treated effluent and a longer column service time. The percentage removal of MB was calculated, and it shows that a 4 cm bed depth has 82.49%, the highest compared to 3 and 2 cm, at 80.93% and 49.05%, respectively. This indicates that a higher bed depth is more effective at absorbing more MB than a lower bed depth.

Effect of Flow Rate on Breakthrough Curve

The influent MB concentration and bed depth were held constant at 50 ppm and 3 cm, respectively, to study the effect of flow rate on MB removal. Column operation was then performed at three different flow rates: 3, 4, and 5 mL/min. The results obtained were plotted for breakthrough curves as shown in Figure 4.

As shown in Figure 4, the adsorption bed was exhausted within 1 to 3 hours after breakthrough, indicating that the adsorption zone was almost saturated at the time of exhaustion. These types of breakthrough curves are highly desirable. The breakthrough times (corresponding to C/C0 = 0.200) were 63.40, 13.75, and 0.5 minutes at flow rates of 3, 4, and 5 mL/min, respectively. The exhausted times (corresponding to C/C0 = 0.995) were 188, 118.75 and 46 minutes, respectively. The corresponding volumes of wastewater

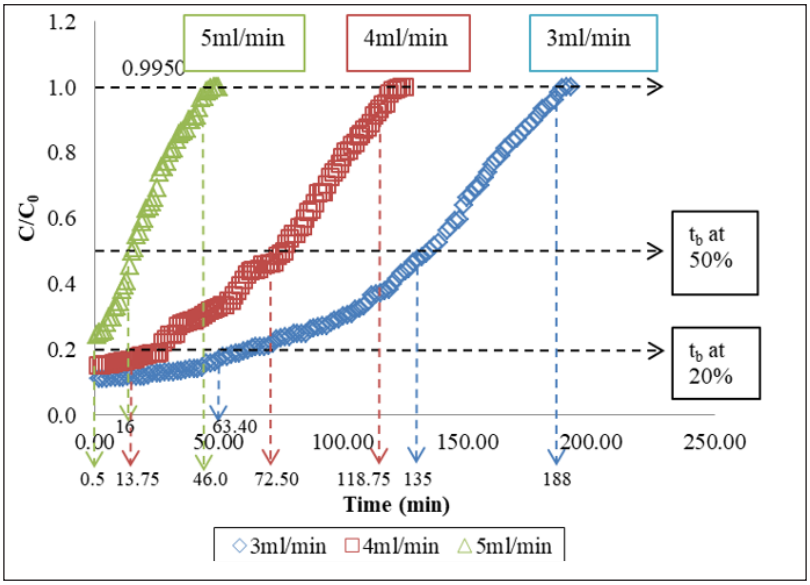

Figure 4. Flow rate effect on the breakthrough curve on durian peels MB adsorption at c0 = 50 mg/l and 3 cm bed depth

treated at the breakthrough point were 317, 68.75, and 2.5 mL, respectively, and at the exhaust point were 940, 593.75, and 230 mL, respectively.

Figure 4 indicates that the higher flow rate reached breakthrough faster than the lower flow rate, and the column exhaustion time increased significantly with reduced flow rate. At a low influent rate, the contact time between MB and durian peel was longer, resulting in higher MB ion removal in the column. It was observed that the breakthrough curves became steeper as the flow rate increased. The column system's exhaustion point increased at lower flow rates. Although MB adsorption was rapid, diffusion effects were reduced due to insufficient dye residence time in the column at higher flow rates. Hence, lower flow rates were desirable for effective MB removal in the column design.

In mass transfer fundamentals, capacity can be inferred from the diversity of breakthrough curve slopes, along with adsorption capacity. According to Ko et al. (2000), the mass transfer rate increased with flow rate, increasing the amount of dye adsorbed onto the adsorbent bed; therefore, the column is exhausted faster at higher flow rates. As shown in Table 2, the column reached the exhaustion condition faster at 5 mL/min (within 46 minutes) than at 3 mL/min (188 minutes). The percentage removal of MB was calculated, and it shows that a flow rate of 3 mL/min yields 89.56%, the highest compared to 4 and 5 mL/min, at 80.93% and 44.37%, respectively. This indicates that a lower flow rate is more effective at absorbing more MB than a higher flow rate.

Table 2
Column breakthrough parameters on flow rate

Flow Rate	HRT (min)	T_b (min)	V_b (mL)	t_{exh} (min)	V_{exh} (mL)	% Removal
3 mL/min	1.7674	63.4	317	188	940	89.56
4 mL/min	1.3255	13.75	68.75	118.75	593.75	80.93
5 mL/min	1.0604	0.5	2.5	46	230	44.37

Table 3
Thomas model parameters

Dye	Adsorbent	Parameter		K_{Th} (mL mg-1 min-1)	q_0 (mg g-1)	R^2
MB	DP	bed depth	2 cm	0.2011	79.113	0.9969
			3 cm	0.0399	264.343	0.9968
			4 cm	0.0031	821.816	0.9926
		flow rate	3 mL/min	0.0092	1268.712	0.9943
			4 mL/min	0.0399	264.343	0.9968
			5 mL/min	0.3910	17.584	0.9968

Thomas Model

Generally, to determine the solid-phase concentration corresponding to the adsorbent's maximum adsorption capacity and to obtain a concentration-time profile or breakthrough curve, column experiments should be conducted (Yan et al., 2001). According to Ibrahim (2010), a widely used model for regulating the ultimate solid-phase concentration of solute on the sorbent and the rate constant in an adsorption column is the Thomas model, developed by Thomas.

As tabulated in Table 3, the number of R2 values was significantly high for all column system simulations at a range of 0.99-1.00. This indicates that all breakthrough curves were well predicted by the Thomas model, as shown in Figures 5 and 6. The phenomenon of axial dispersion was reduced by the uniform size of the adsorbent, and the dye solution was distributed evenly, making the Thomas model suitable.

The Thomas model parameters obtained were tabulated in Table 4, where kTh and q0 represent the Thomas rate constant and the equilibrium MB uptake, respectively. Column adsorption capacity was recorded as q0, and the operation with the highest bed depth yielded the largest q0, 821.816 mg g-1 at a bed depth of 4 cm. As for the flow-rate effect, the highest q0 was recorded at the lowest flow rate, 3 mL/min, at 1268.712 mg g-1.

As the flow rate increased, q0 decreased, while kTh increased. As the bed depth increased, the value of q0 increased significantly, while the value of kTh decreased significantly. Thus, a lower flow rate and a higher bed depth increased MB adsorption on durian peels.

Table 4
Yoon-Nelson model parameters

Dye	Adsorbent	Parameter		K_{Th} (mL mg-1 min-1)	τ (min)	$t_{50\%}$exp (min)	R^2
MB	DP	bed depth	2 cm	0.00020	26.25	26.25	0.9942
			3 cm	0.00008	72.50	76.25	0.9970
			4 cm	0.00006	96.25	97.50	0.9951
		flow rate	3 mL/min	0.00005	133.60	135.27	0.9961
			4 mL/min	0.00008	72.50	76.25	0.9970
			5 mL/min	0.0029	16.00	15.00	0.9966

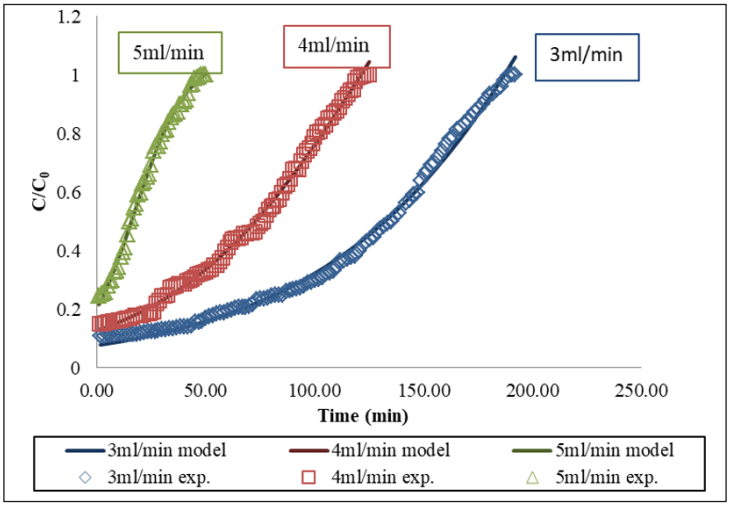

Figure 5. Nonlinear Thomas model curves on various bed depths

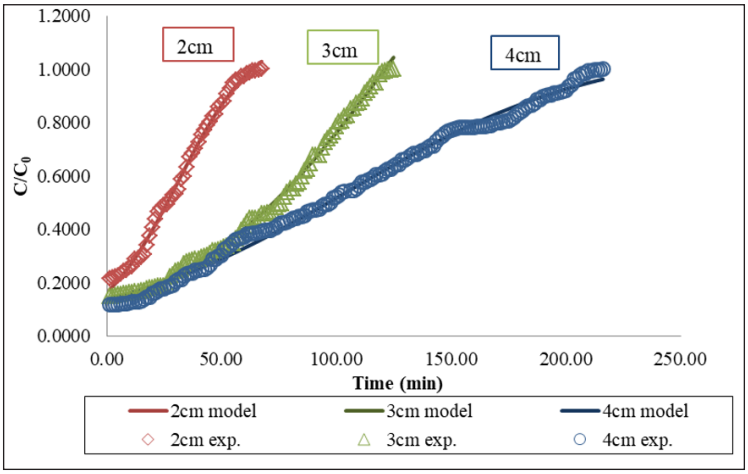

Figure 6. Nonlinear Thomas model curves on various flow rates

Yoon-Nelson Model

This uncomplicated theoretical model was formulated by Yoon-Nelson to investigate column breakthrough characteristics. Assuming that the reduction in the probability of each absorber being adsorbed is comparable to the feasibility of adsorption and breakthrough in the adsorbent, the Yoon-Nelson model is very simple in form (Xu et al., 2013). This model is also reliable, as is the Thomas model, since it provided an acceptable fit to the empirical data. Additionally, this model is expected to yield identical R2 values, since it predicts similar uptake capacity and Ct/Ci values for a given data set.

As tabulated in Table 4, the number of R2 values was significantly high for all column system simulations at a range of 0.99-1.00. This indicates that all breakthrough curves were well predicted by the Yoon-Nelson model, as shown in Figures 7 and 8. In Table 4, the Yoon-Nelson parameters were denoted as kYN (the Yoon-Nelson rate constant), while the time required for 50% breakthrough was denoted by τ. The time required for 50% breakthrough was highest at the lowest flow rate, 133.60 minutes at 3 mL/min, compared to 72.50 and 16 minutes at 4 and 5 mL/min, respectively. This model prediction was like the 50% breakthrough time from the bed-depth experimental findings, which were 135, 72.50, and 16 minutes at flow rates of 3, 4, and 5 mL/min, respectively.

The significant outputs from the Yoon-Nelson model were the regression coefficient value (R2) and the time required for 50% breakthrough (τ). The nonlinear plots of the Yoon-Nelson model yielded the best fit, with R2 values of 0.99-1.00 across all column systems. The R2 values were important in showing that the experimental data were well predicted. The τ values were compared with the experimentally obtained time to 50% breakthrough.

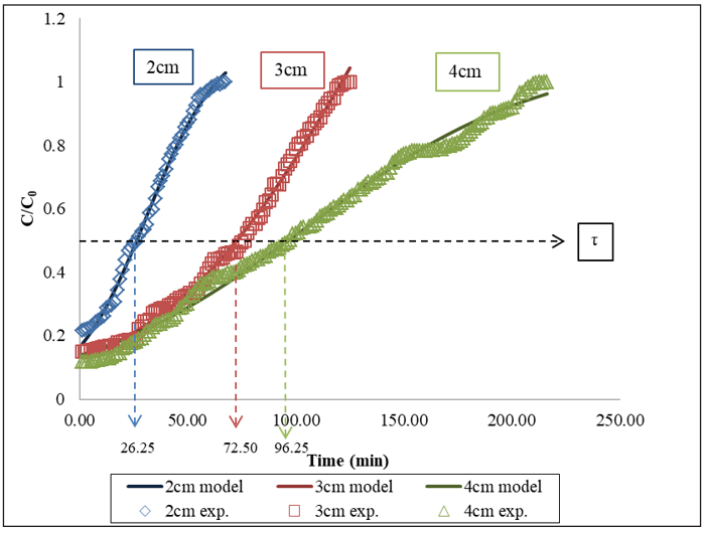

Figure 7. Nonlinear Yoon-Nelson model curves on various bed depths

As presented in Table 4, the τ values for the bed depth study were 26.25, 72.50, and 96.25 minutes for bed depths of 2, 3, and 4 cm, respectively. Similar values were recorded from experimental findings: 26.25, 72.50, and 92.5 minutes for bed depths of 3, 5, and 7 cm, respectively. As for the flow rate study, the τ values matched the experimental flow rate findings: 133.60, 72.50, and 16 minutes for 3, 4, and 5 mL/min, respectively.

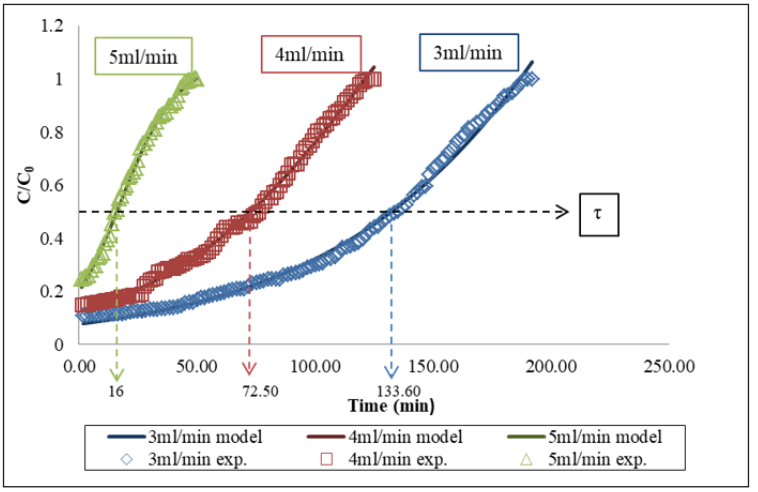

Figure 8. Nonlinear Thomas Model curves on various flow rates

CONCLUSION

In this study, two parameters were considered in the column design. The main parameters in this column experiment are bed depth and flow rate. The objectives of this study have been successfully attained, as durian peels are effective as absorbents for removing dyes from wastewater. This is evident from the results, which show that the breakthrough curves of each experimental data set are very similar to the suggested or standard pattern for breakthrough curves in adsorption studies. The other objective of this study has also been achieved: simulating the experimental data using the column mathematical models implemented here, namely the Thomas and Yoon-Nelson models. From this study, it was concluded that:

Bed depth parameters can be improved by increasing the adsorbent bed depth in the column design. This action will result in a higher amount of MB volume that can be removed. This is because the higher the bed height, the greater the adsorbent's capacity to adsorb MB.

As for flow rate parameters, they contrasted with bed depth: the lower the flow rate used in the column designs, the greater the MB volume removed. Hydraulic retention time (HRT) is highest at the lower flow rate, indicating that the contact time between the

adsorbent and the dye is longer. This allows molecules and particles to adhere, thereby increasing the volume of MB removal.

The mathematical models, Thomas, can be well equipped with experimental data once applied to them. It was a convenient way to determine whether the experimental data were studied efficiently. The coefficient of determination (R2) values ranged from 0.99 to 1.00. This demonstrates that the dynamic model fitted well with experimental breakthrough curves. As Nainamalai et al. (2018) noted, the R2 values ranging from 0.797 to 0.970 indicate that the model fits well.

As for the Yoon-Nelson model application, it shows that the model fitted experimental data as well. The coefficient of determination (R2) values ranged from 0.99 to 1.00. This demonstrates that the dynamic model fitted well with experimental breakthrough curves. As Nainamalai et al. (2018) noted, the R2 values ranging from 0.797 to 0.970 indicate that the model fits well. In addition, the τ value obtained from the Yoon-Nelson model represents the time to 50% breakthrough and was nearly equal to the 50% breakthrough time from the experimental findings for both bed depth and flow rate studies.

Generally, durian peels, as an agricultural solid waste, can be found abundantly disposed of mostly in the countryside. Because of that, it was inconvenient to implement an economical method for using durian peels as an adsorbent without chemical processing. The industrial sector that currently uses dyes for discharge can implement this design as one way to filter before directly discharging into waterways. A pre-treatment process may be implemented to enhance performance and meet the industry standard.

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