

Coconut Husk Adsorbent for the Removal of Methylene Blue Dye from Wastewater

Hasfalina C. B. Man,^{a,*} Christopher O. Akinbile,^{a,b} and Chin X. Jun^a

A study to assess the efficiency of coconut husks (CHs) in removing methylene blue (MB) dye from wastewaters in Malaysia was carried out. A fixed bed column adsorber was set up using flow rates of 40 and 80 mL/min, and the adsorbent (CH) was prepared using the base treatment method with NaOH as activating agent. Three different column bed depths (10, 20, and 25 cm) and unit weights of adsorbent (103, 213, and 260 g) were used. Two models, the bed depth service time (BDST) and Thomas models, were used to validate the adsorption capacity results and breakthrough curve. Changing the bed depth from 20 to 25 cm did not result in a significant change in adsorption capacity, therefore a 20-cm bed depth is recommended as the most efficient. Similarly, adsorption capacity increased as flow rates increased from 40 to 80 mL/min, indicating that a flow rate of 80 mL/min yielded optimum efficiency. The two models also provided predictions with good fits of the bed depth effect, the adsorption capacity, and the breakthrough curve of CH for MB removal.

Keywords: Coconut husk; Methylene blue; Adsorption; Wastewater; Models; Breakthrough curve; Malaysia

Contact information: a: Department of Biological & Agricultural Engineering, Universiti Putra Malaysia (UPM), Malaysia; b: Department of Agricultural & Environmental Engineering, Federal University of Technology, Akure, Nigeria; *Corresponding author: has@eng.upm.edu.my

INTRODUCTION

Water pollution has become a serious environmental problem around the world and is caused by the addition of chemical, physical, or biological substances in certain concentrations, either naturally or of man-made origin (Metcalf *et al.* 2003; Forgacs *et al.* 2004). Many Malaysian industrial sectors, such as the textile, paper, paint, pharmaceuticals, food, leather, cosmetics, tannery, printing, and plastics industries, use wide varieties of dye to color their products (Wong *et al.* 2013). Considering the volume discharged and the effluent composition, wastewater from the textile industry has been declared one of the major wastewater pollution sources in Malaysia. Dyes have been extensively used in many industries (including textile, leather tanning, paper production, food technology, and medicine). There are more than 100,000 types of commercially available dyes, and more than 700,000 tons of dyes are produced annually (Ferrero 2007). Of all available dyes, methylene blue (MB) is the most commonly used in industries such as textiles (dyeing cotton, wood, and silk) and pharmaceuticals. MB can cause eye burns and permanent injury to the eyes of both humans and animals (mostly aquatic) and is a monoamine oxidase inhibitor (MAOI) that inhibits the activity of the monoamine oxidase enzyme family (Bellir *et al.* 2010). MB dye (C₁₆H₁₈N₃SCL) is also accepted as a model compound for adsorption of medium sized organic molecules (Karacetin *et al.* 2014; Aci *et al.* 2008). Although it can be used as a pharmaceutical drug, it can also cause short

periods of rapid or difficult breathing, and ingestion causes a burning sensation and sometimes nausea, vomiting, profuse sweating, mental confusion, and methemoglobinemia (Bulut and Aydin 2006). Treatment of effluent containing MB is of paramount interest because of its negative impacts on receiving bodies of water. To treat wastewater infested with MB, two main physical processes are conventionally employed: adsorption and membrane separation (Brodin and Theliander 2012).

Adsorption is the most common and widely used method and is considered the most effective, economical way to remove MB by decreasing its concentration. Membrane separation involves relatively expensive techniques such as ultrafiltration, reverse osmosis, ozonation, and nanofiltration. Hubbe *et al.* (2012) remarked that adsorption experiments have been conducted to study the effect of the initial dye concentrations of adsorbent in solutions on the rate of dye adsorption onto adsorbent. Such experiments are usually carried out at a fixed adsorbent dose and at different initial dye concentrations of adsorbent for different time intervals and at a fixed pH and agitation speed. However, they further noted that the percentage removal generally will decrease with a decrease in initial concentrations. Most commercial dye removal systems for industrial wastewater use activated carbon as an adsorbent (Hammed *et al.* 2009). However, the production cost of activated carbon adsorbent is high since it requires a furnace and a burning process. The rate of combustion is directly proportional to carbon dioxide production, a significant greenhouse gas. Similarly, the rate of regeneration limits its utilization apart from the costly production (Wang and Li 2013). Recently, numerous approaches have been explored to determine a cheap and effective adsorbent derived from a variety of raw materials waste such as agro-waste.

In the last few years, research has focused on several agricultural lignocellulose-based adsorbents because they are renewable, biodegradable, eco-friendly, and cheap (Ferrero 2007). These materials are used to make useful, value-added adsorbents for wastewater treatment, especially in Malaysia (Kumar and Kumaran 2005). The material used to make the cheapest and most readily-available adsorbent is coconut husk. Coconut husk (CH) is the mesocarp of the coconut which makes up 33 to 35% of the husk (MOA 2009). Many studies of the use of agricultural by-products such as rice straw, rice husk, oil palm fibre, and rubber wood sawdust as adsorbent materials in water treatment have been carried out, but coconut husk seems to be the most preferred, as 60% of the husk is cellulose and lignin (Sivapragasam 2008). The hydroxyl groups in these two polymers provide sites for dye adsorption (Neto *et al.* 2011; Wong *et al.* 2013). Coconut is considered the fourth most important commodity crop in Malaysia after oil palm, rubber, and paddy rice in terms of total planted area (MOA 2009). The coconut industry contributes very little to the overall Malaysian economy, accounting for approximately 0.08% of its exports earnings in 2006 (MOA 2009). Malaysia has a total coconut plantation land area of 109,185 hectares managed by about 80,000 farmers and producing approximately 382 million tons of coconut 2007 (MOA 2009). The negative impact of the husk and shell by-products on the environment would be alarming if proper disposal and treatment systems were not put in place. Thus, adding value by converting the husk to adsorbent has become a viable option.

The objective of the present study was to investigate the adsorption capacity of untreated coconut husk as an adsorbent for removing methylene blue from aqueous solution using the fixed bed column method. The tools used to determine the adsorption capacity of the CH include the BDST model for constant volume, the Thomas model for constant rate, as well as breakthrough and exhaustion points from the experimental data. Most biosorption

studies have been carried out using a batch sorption system with a focus on bio-adsorbent properties. These studies have used rice husk, wheat shells, mango seed kernel, and palm kernel fibre, but in practical terms, biosorption on a large scale is usually carried out continuously in a fixed bed column system (Kumar and Kumaran 2005). Only one fixed bed column adsorption method has been used in the adsorption of methylene blue by coconut husk in previous studies (Tan *et al.* 2008; Teixeira *et al.* 2013). The need to determine the adsorption capability of coconut husk on MB using a fixed column necessitated this adsorption study.

EXPERIMENTAL

Adsorbent Preparation and Characterization

Coconut husks (CHs) collected from a coconut product factory in Kluang, Johor were used as an adsorbent to remove dye molecules (methylene blue, MB) from aqueous solution. CHs were processed *via* cutting and sieving through 2.0-, 0.6-, and 0.15-mm plates using a sieve machine. Products between 0.15- and 0.6-mm in size were used in this experiment. All experiments were carried out at room temperature (25 ± 2 °C) and atmospheric pressure (1 atm). The adsorbate, a solution of methylene blue (MB), was prepared using base treatment method with 0.5 M of NaOH as activating agent and stirring at room temperature as described by Wang and Li (2013), and dye concentrations were determined using an DR/4000U Spectrophotometer (HACH, Iowa, US). Characterization of the adsorbent was done. Average values of the three replicate measurements of the integrated absorbance were presented in the results. CH was used as a methylene blue adsorbent in the fixed bed column process. The parameters studied include the adsorptive equilibrium as functions of concentration, dosage, particle size, and temperature. The effect of bed depth and the flow rate of the aqueous phase on column performance were also evaluated. A series of fixed bed experiments was undertaken and the results were applied to a bed-depth/service time (BDST) model and a Thomas model for column adsorption. The breakthrough data was analyzed using bed depth/service time (BDST) and Thomas models. The BSDT model is also known as the Bohart and Adam model, as described by Singh and Pant (2006),

$$t = \frac{N_0 Z}{C_0 v} - \frac{1}{k_a C_0} \ln \left(\frac{C_0}{C_b} - 1 \right) \quad (1)$$

where t is the service time at the breakthrough point (5%) in h, N_0 is the adsorption capacity per unit volume of bed in mg/L, Z is the depth of the adsorbent bed in cm, C_0 is the influent of initial solute concentration in mg/L, v is the linear flow rate in cm/h, k_a is the rate constant of adsorption in L/mg·h, and C_b is the effluent concentration at the breakthrough point (5%) in mg/L.

The Thomas model, often used to determine the dynamic characteristics of the biosorption process in fixed bed columns, as described by Calero *et al.* (2009), is,

$$\frac{C}{C_0} = \frac{1}{1 + \exp \left(\frac{k_{TH}}{F} (Q_0 M - C_0 V) \right)} \quad (2)$$

where k_{TH} is the Thomas rate constant in mL/mg·h, Q_0 is the maximum concentration of solute adsorption per unit mass in mg/g, M is the amount of adsorbent in the column in g,

V is the effluent volume in L, F is the flow rate in mL/h, and C is the effluent concentration in mg/L.

The linear form of the Thomas model is expressed below:

$$\ln\left(\frac{C_0}{C} - 1\right) = \frac{k_{TH}Q_0M}{F} - \frac{k_{TH}C_0V}{F} \quad (3)$$

The kinetics coefficient k_{TH} and the adsorption capacity of the column Q_0 can be determined with a non-linear regression graph of $\ln(C_0/C - 1)$ against t at a given flow rate.

Design, Development, and Operations of a Fixed-Bed Column Adsorber

A fixed-bed column adsorber made from white PVC with an 11-cm diameter and 60-cm height was developed and used for the experiments. Glass fiber nets were placed at the bottom of the column to prevent the adsorbent from leaching into and clogging the drainage. The nets were also placed on top of the column to increase the distribution of solution onto the adsorbent surface and maintain a constant flow rate. The top of the column was left open to the atmosphere to maintain the internal pressure of the column near atmospheric pressure, similarly as in the study of Calero *et al.* (2008). A peristaltic pump was installed and adjusted to flow rates ranging between 40 and 80 mL/min with a fixed initial concentration of 75 mg/L during all experiments. A schematic diagram of the experimental set-up used is shown in Fig. 1. As for the characterization, the adsorbent (CH) was packed into the column at different bed depths of 10, 20, and 25 cm, while the unit weights of the adsorbent tested at each depth were 103, 213, and 260 g. The initial concentration of the dye solution was 75 mg/L, and the solution was fed downward into the column using a peristaltic pump (RP1000, EyelaWorld, Japan) with breakthrough (t_b) and exhaustion (t_e) times determined in all the bed depths and at the two flow rates of 40 mL/min and 80 mL/min, respectively. The treated samples were immediately collected from the exit at fixed time intervals and measured for their remaining dye content to identify the bed breakthrough and exhaustion times. The column was operated until the concentration of dyes in the effluent reached 95% of the initial concentration and sampling was carried out hourly until a breakthrough curve was obtained.

The equilibrium adsorption isotherm is very important in the design of an adsorption system. For solid-liquid systems, several isotherms are available to elucidate the mechanism of the adsorption process but the one used in this study is the Langmuir isotherm equation which is given, according to Ho *et al.* (2002) as,

$$q_e = q_m k_a C_e / (1 + k_a C_e) \quad (4)$$

where q_e is the amount of dye adsorbed per unit mass at equilibrium (mg/g), q_m is the maximum possible amount of dye that can be adsorbed per unit mass adsorbent (mg/g); C_e is the concentration of sorbate in the solution at equilibrium (mg/L); and k_a is the sorption equilibrium constant. The linearised form of Eq. 4 can be expressed as:

$$C_e/q_e = 1/(q_m k_a) + C_e/q_m \quad (5)$$

Statistical Analysis of Data

The results obtained were subjected to statistical analysis using Microsoft Excel spreadsheet (ANOVA), Statistical Package for the Social Sciences (SPSS) Version 15.1

software, multiple linear regression, and Least Square Difference (LSD) at the 95% significance level.

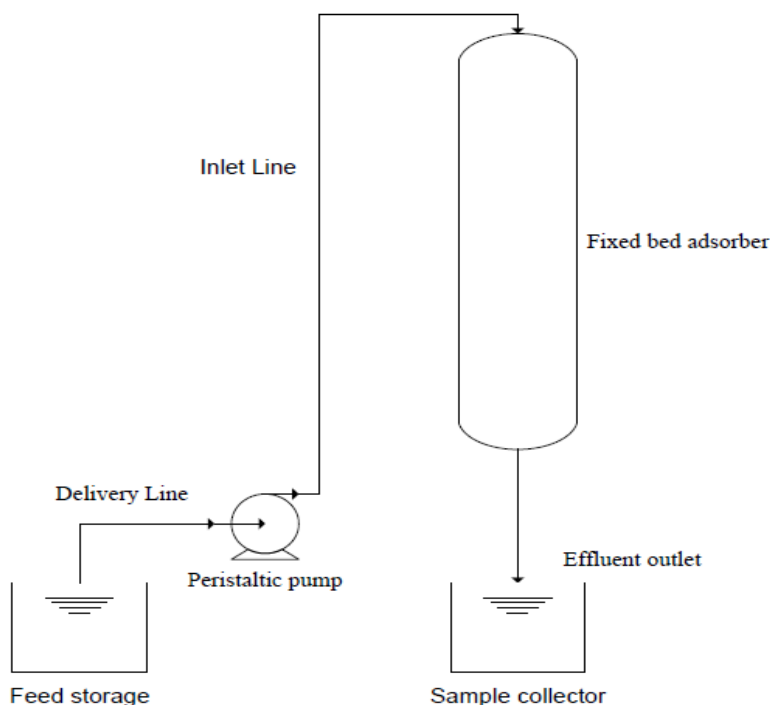


Fig. 1. Schematic diagram of fixed-bed column adsorber

RESULTS AND DISCUSSION

Effect of Bed Depth and Contact time on Breakthrough Curve

Dye adsorption in the fixed bed column exhibited a positive relationship with the quantity of adsorbent in the column, indicating that with increases in the bed depth and the volume of aqueous dye solution, the quantity of dye removed increased. The breakthrough curve profiles for MB adsorption at different bed depths with flow rates at 40 and 80 mL/min are presented in Figs. 2 and 3, respectively. The breakthrough time (t_b) and exhaustion time (t_e) increased with increasing bed depth (Table 1). The S-shape curve plotted from t_b to t_e decreased as the bed depth increased from 10 to 25 cm, indicating that the adsorption capacity of CH on MB increased slightly with increasing column bed depth. The increasing adsorption capability of CH observed in this study was probably due to the increase in the surface area of the adsorbent and the extended contact time with the MB solution. Similar observations have been reported by several other researchers (Wang *et al.* 2014; Vadivelan and Kumar 2005; Vijayaraghavan and Prabu 2006; Amarasinghe 2011). Figures 2 and 3 show that it took a longer time for CH to be exhausted with greater bed depth. An increase in the exhaustion time (t_e , the time taken for the effluent to reach 95% the concentration of the MB influent) was observed when the bed depth was increased from 10 to 25 cm.

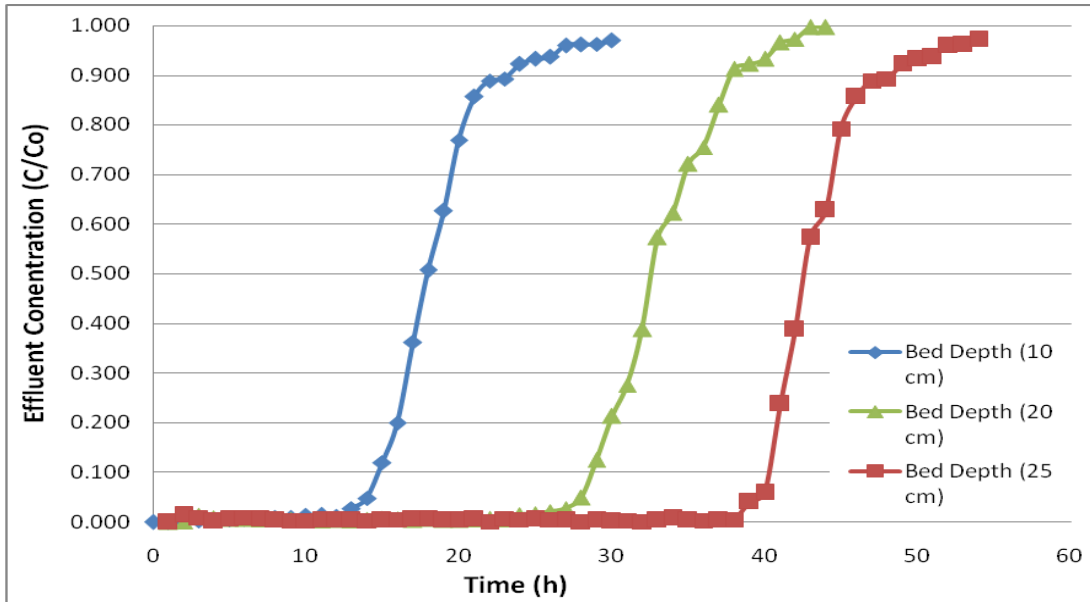


Fig. 2. Effect of bed depth on the breakthrough curve at 40 mL/min flow rate

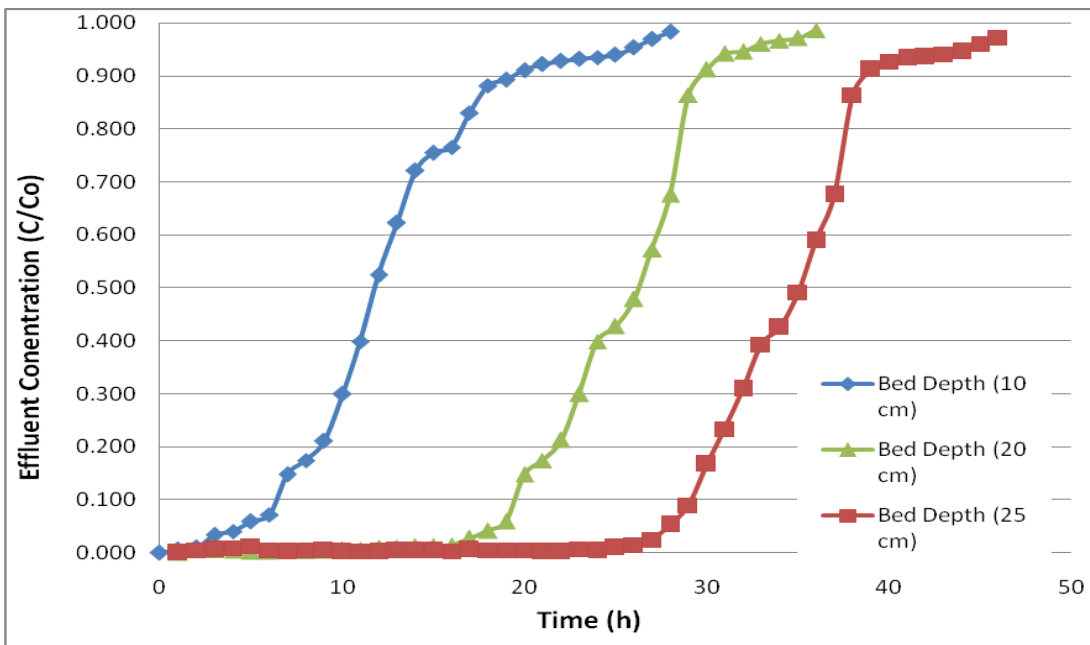


Fig. 3. Effects of bed depth on the breakthrough curve at 80 mL/min flow rate

Table 1. Experimental Constants of BDST Model for MB Adsorption by Coconut Husk ($C_0 = 75 \text{ mg/L}$)

Bed Depth Z (cm)	40 mL/min Flow Rate			80 mL/min Flow Rate		
	t_b (h)	t_e (h)	R^2	t_b (h)	t_e (h)	R^2
10	14.03	26.57	0.9761	4.49	25.76	0.9973
20	24.00	39.55		13.00	31.28	
25	38.49	50.57		26.89	43.26	

Since only two flow rates were used in this study, whenever exhaustion time was attained with the CH, only 5% of MB from aqueous solution was removed and the remainder was left to flow out of the column. For 40 mL/min flow rate, the exhaustion time for CH increased from 26.57 to 50.57 h, whereas for 80 mL/min flow rate, the exhaustion time increased from 25.76 to 43.26 h. The increase in MB removed by CH was achieved at the beginning of the process, and immediately following the breakthrough time, the concentration of dye in the effluent rapidly increased. The shape and gradient of the breakthrough curves were formed slightly differently than the bed depth curves. This is in agreement with the findings of Chen and Liu (2012) and Allen and Koumanova (2005).

The data collected were analyzed using the BDST model shown in Eq. 1. Figure 4 shows the linear relationship between the service time and the bed depth on MB adsorption using coconut husk. The linear regression coefficients of determination (R^2) were 0.97 and 0.90 at 40 and 80 mL/min, respectively. This clearly validates the use of the BDST model to predict the adsorption of MB by the coconut husk. There was a positive relationship between the service time and the bed depth. The adsorption capacity per unit volume of bed, N_0 , and adsorption rate constant, K_a , were computed from the slope and intercept of the BDST model with the assumption that the initial concentration of MB in the aqueous solution, C_0 , and the linear velocity, v , remained constant throughout the entire column. The rate adsorption rate constant, K_a , is a measure of the transfer rate of dye from the fluid phase to a solid phase; that is, the rate at which the particles of dye form bonds with the adsorbent. For a constant flow rate of 40 mL/min, the N_0 and K_a were 858,737 mg/L and 0.0105 L/mg·h, respectively, and for 80 mL/min, N_0 and K_a were 1,520,723 mg/L and 0.0024 L/mg·h, respectively. The difference in N_0 between the two flow rates was 661,986 mg/L. Similar findings were reported by Chowdhury *et al.* (2015) and Wang *et al.* (2014).

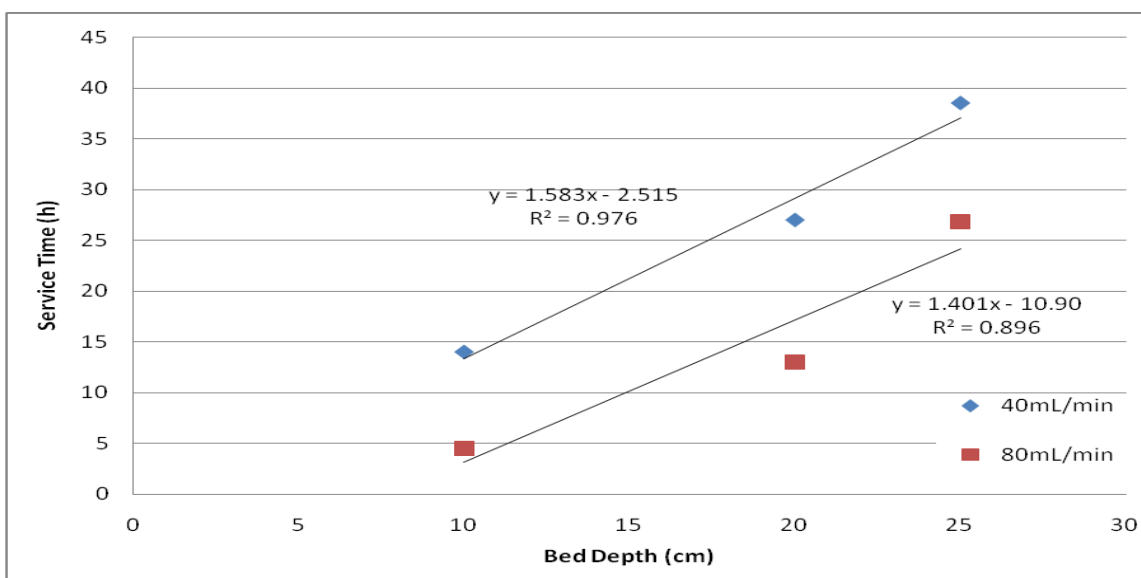


Fig. 4. BDST model plot for MB adsorption on coconut husk (CH) with $C_0 = 75$ mg/L

Effect of Flow Rate and Adsorption Capacity of Adsorbent on Breakthrough Curve

The capacity of CH to remove MB from aqueous solutions at different flow rates is shown in Table 2. The breakthrough curves plotted are shown in Figs. 5, 6, and 7. With increases in flow rate, the breakthrough and exhaustion times were shorter. The MB adsorption per unit adsorbent mass decreased slightly with increases in the flow rate at the same bed depth. Several researchers (Tan *et al.* 2008; Mondal 2009) have observed similar patterns in their findings, indicating that the column adsorption method performed more effectively at the lower flow rate. Results from other studies show that when the flow rate increases, the adsorption capacity marginally decreases (Ofomaja 2007; Hubbe *et al.* 2012; Danish *et al.* 2013). The adsorbate leaves the column without sufficient time to diffuse into the pores of the adsorbents, resulting in earlier breakthrough and exhaustion times and a steeper breakthrough curve, as shown in Figs. 5, 6, and 7. To overcome the short contact time, a reduction in the flow rate of adsorbate into the column should be made, allowing the dye molecules more time to react with or diffuse into the pores of the adsorbent. Yagub *et al.* (2014) reported similar findings in their work.

Table 2. Constant of Thomas Model for MB Adsorption by CH ($C_0 = 75$ mg/L)

F (mL/min)	Bed depth (cm)					
	10		20		25	
	k_{TH} (L/mg·h)	Q_0 (mg/g)	k_{TH} (L/mg·h)	Q_0 (mg/g)	k_{TH} (L/mg·h)	Q_0 (mg/g)
40	0.0054	33873	0.0034	27316	0.0023	33126
80	0.0041	50756	0.0043	42026	0.0031	49111

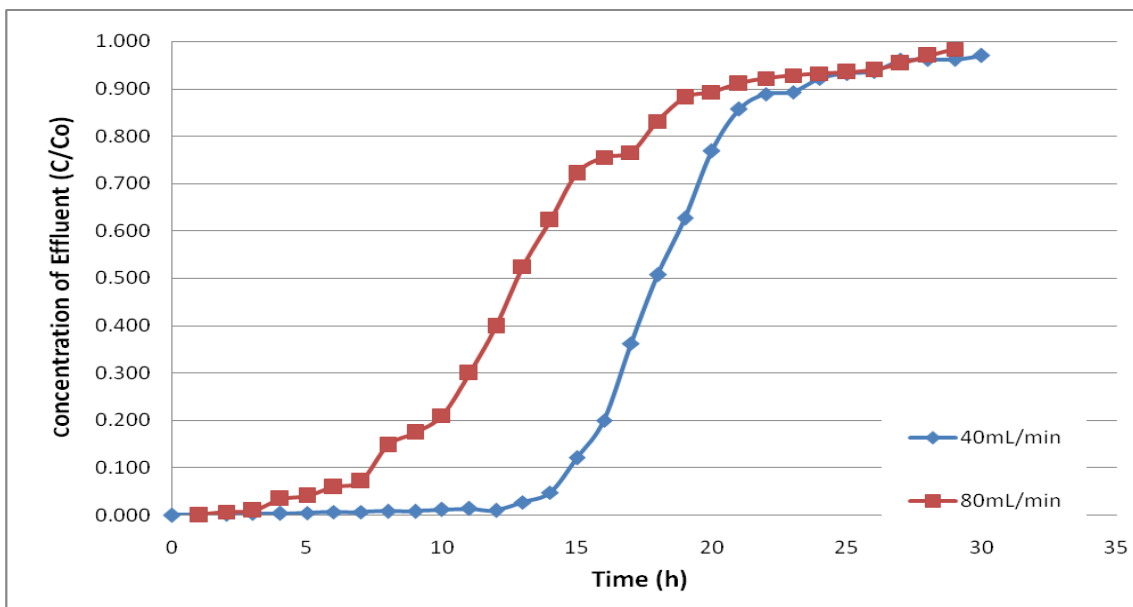


Fig. 5. Effects of flow rate on the breakthrough curve at 10 cm bed depth

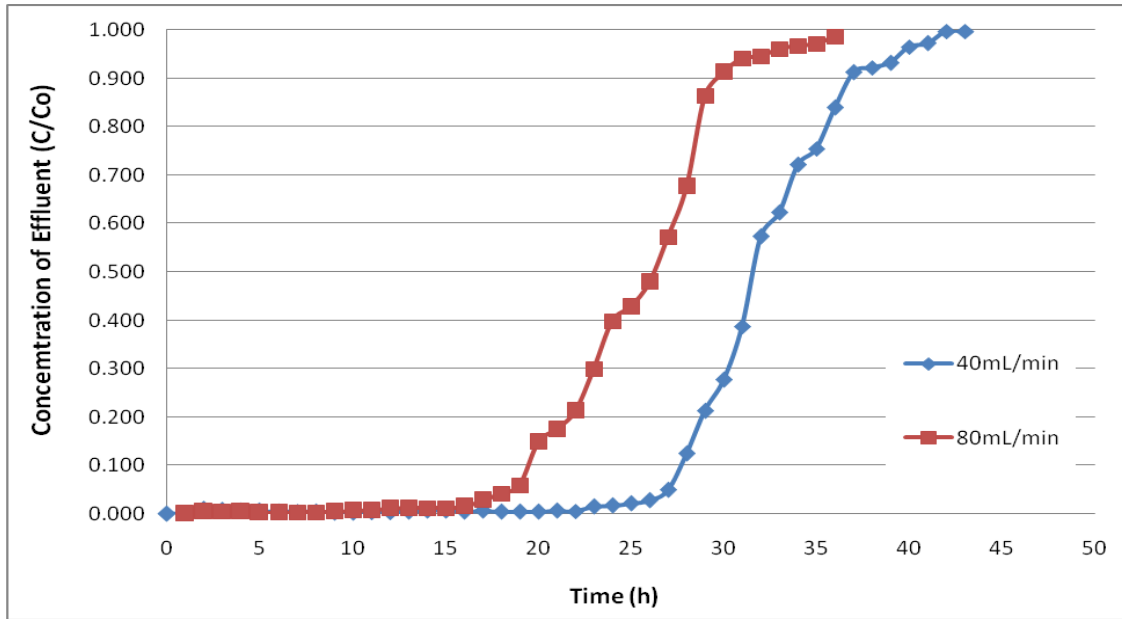


Fig. 6. Effects of flow rate on the breakthrough curve at 20 cm bed depth

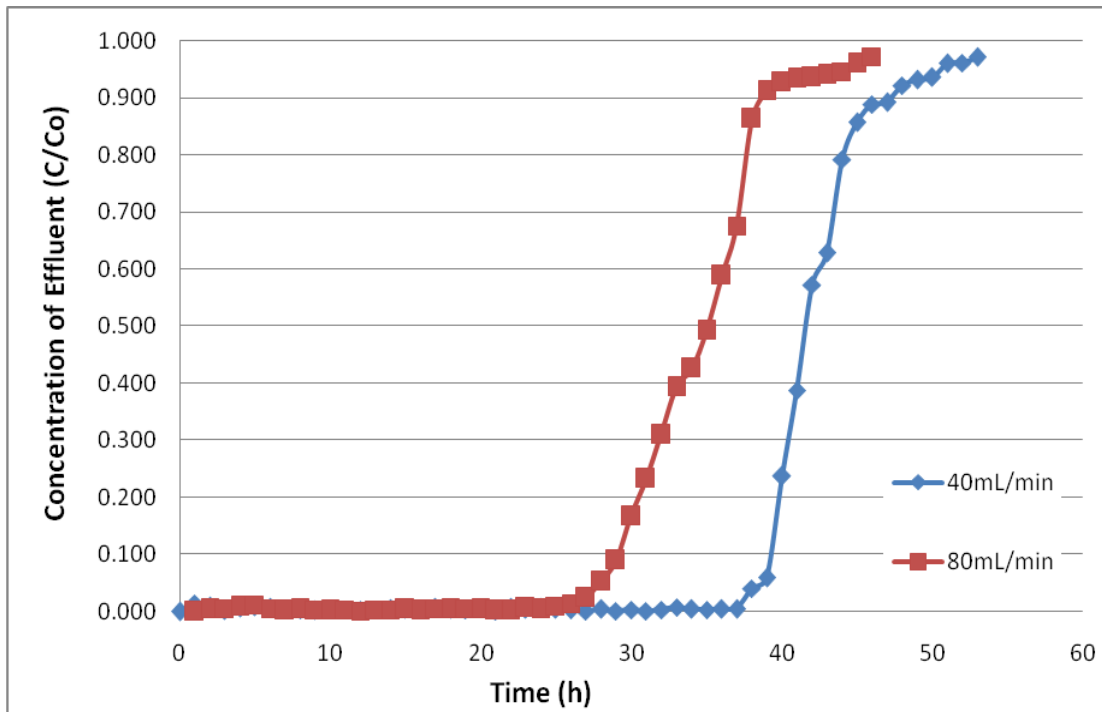


Fig. 7. Effects of flow rate on the breakthrough curve at 25 cm bed depth

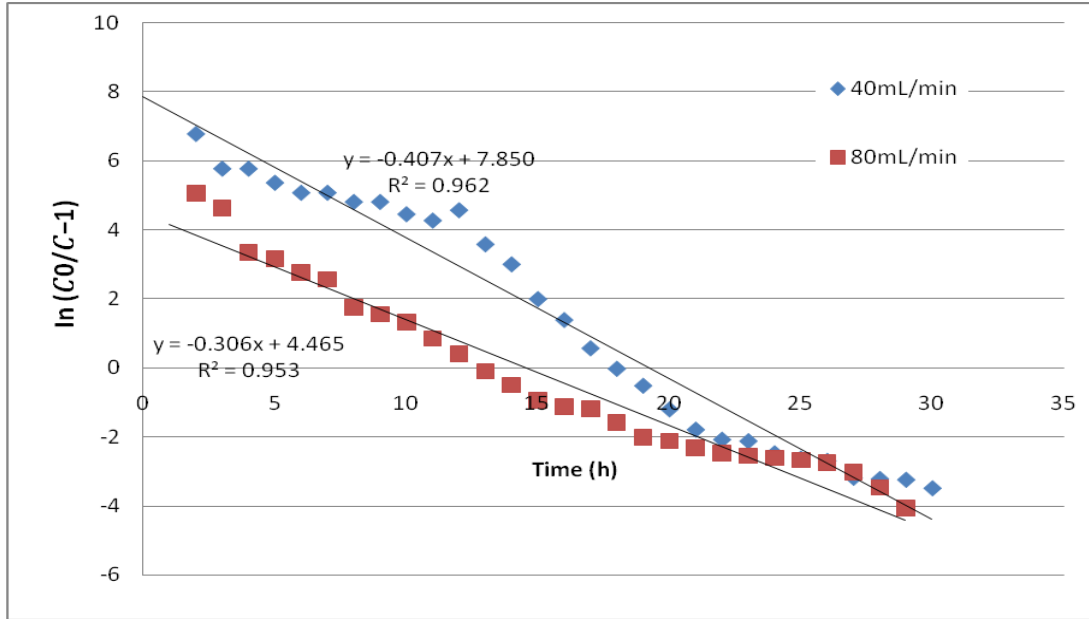


Fig. 8. Thomas model for removal of MB with CH with $C_0 = 75$ mg/L and 10 cm bed depth

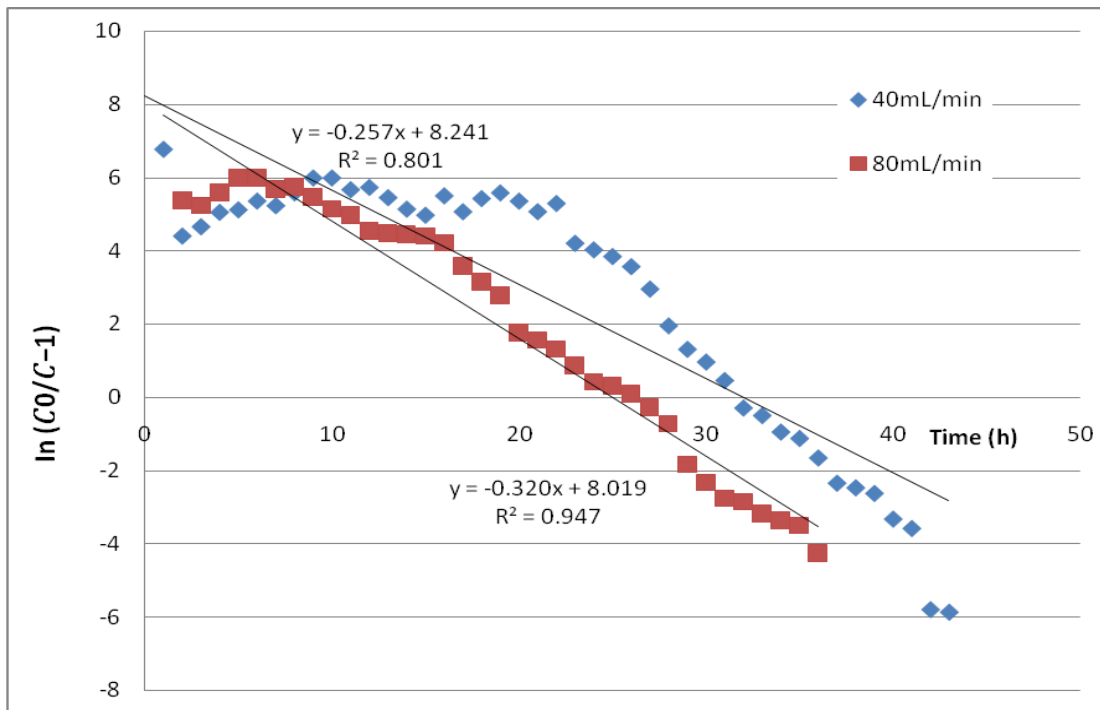


Fig. 9. Thomas model for removal of MB with CH with $C_0 = 75$ mg/L and 20 cm bed depth

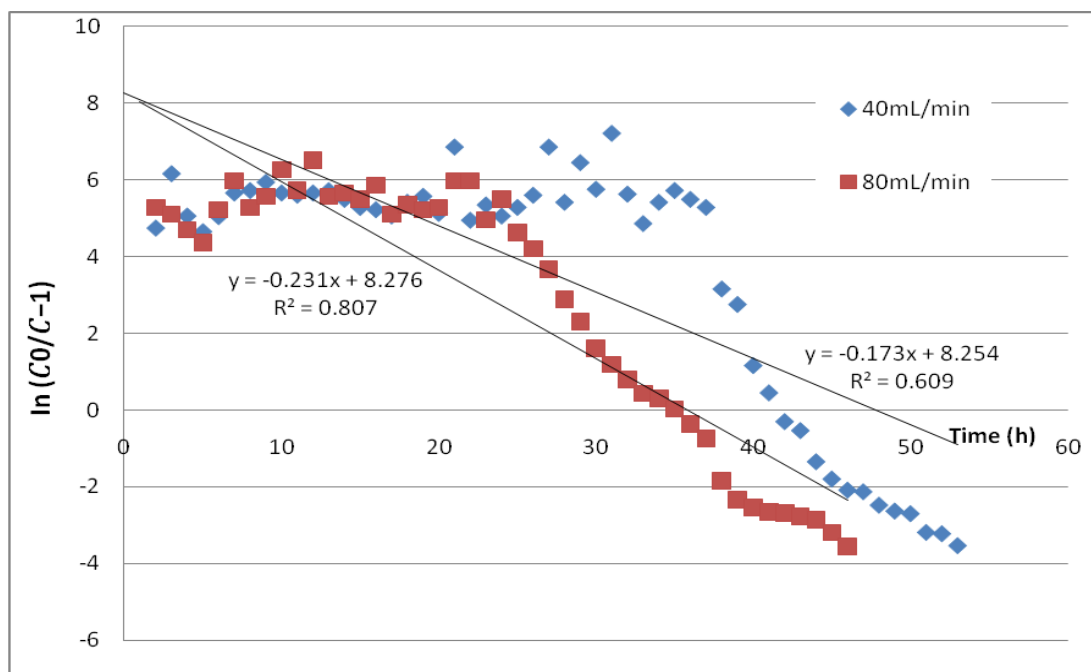


Fig. 10. Thomas model for removal of MB with CH with $C_0 = 75$ mg/L and 25 cm bed depth

The Thomas model was applied to study the effects of flow rate at constant bed depth on the adsorption of MB with CH, as shown in Eq. 3. The results and findings achieved with the Thomas model are presented in Table 2. From the slope and intercept of the graph of $\ln(C_0/C - 1)$ versus t at different flow rates, the rate constant, k_{TH} , and the maximum adsorption capacity, Q_0 , were determined. For the 10-cm bed depth, the two lines plotted using the Thomas model yielded linear regression coefficients (R^2) of 0.96 and 0.95 for 40 and 80 mL/min, respectively, indicating strong and linear negative relationships between $\ln(C_0/C - 1)$ and t . For higher bed height, the linear regression coefficients of the Thomas model graph showed less co-variability between the axes, as shown by the R^2 values in Figs. 8, 9, and 10. The Thomas model results showed that the maximum adsorption capacity changed significantly with increased flow rate. Increasing the flow rate from 40 to 80 mL/min at a constant depth of 10 cm affected the predicted maximum adsorption capacity significantly, changing it from 33,873 to 50,756 mg/g in this study, similar to the findings of Mustafa *et al.* (2014). Higher flow rate yields higher predicted adsorption capacity. Thus, adsorption of MB by a CH adsorbent at a flow rate of 80 mL/min was considered good enough from the context of this study. The exhaustion time at both flow rates did not significantly change; it dropped from 26 to 25 h. The adsorption capacity increased significantly when increasing the flow rate, so 80 mL/min was considered the best flow rate tested in this study. Neto *et al.* (2011) and Oladoja *et al.* (2008) made similar observations in their respective studies.

CONCLUSIONS

1. Coconut husk (CH), a natural, inexpensive, readily-available, environmentally-friendly agricultural waste, is an excellent methylene blue (MB) adsorbent. It could provide an alternative way to adsorb dyes from effluents rather than using costly adsorbents such as activated carbon.

2. CH could be used in industrial wastewater treatment for the textile and fishery industries' MB-containing wastewaters
3. The results of this study suggest that the most suitable fixed-bed column for effective adsorbent capacity has 20-cm bed depth instead of 25-cm bed depth.
4. The flow rate of 80 mL/min was the optimum flow rate applied in this study. It yielded the highest capacity to treat larger volumes of waste.
5. The highest bed capacity was 50,756 mg/g, obtained using 75 mg/L initial MB and an 80 mL/min flow rate at a bed depth of 20 cm.

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