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The Effect of Flowrate on Dye Removal of Jumputan Wastewater in a Fixed-Bed Column by Using Adsorption Model: Experimental and Breakthrough Curves Analysis

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Abstract

One of the traditional arts in Indonesia is Jumputan fabric is produced by using tie and dye technic. The Jumputan wastewater contains organic compounds which can decrease the oxygen content in system. One of the economical and applicable processes to handle the Jumputan wastewater is by adsorption method. The objective of this research was to find out the effect of flowrate of dye wastewater to the adsorption performance of the dye onto activated carbon in a continuous fixed-bed column based on the breakthrough curve parameter. The activated carbon made from betel nuts (Cyrtostachys lakka) with size particle of 60 mesh. The column dimension was 2 inches of inside diameter and 60 cm of height column. The bed height was 10 cm. The feed pumped from the top of column with variation of flowrate of 10, 20 and 30 ml/min. The absorbance of the dye was analyzed by using UV-Vis spectrophotometer. The adsorption column models were analyzed using Thomas, Yoon-Nelson, and Adam-Bohart. The result of this research showed that the dye removal efficiency decreased with the increase in flowrate, which was 61.4%; 56.9%; and 47.6% for 10, 20, and 30 ml/min respectively. Feed flowrate showed a negative effect on the saturation time, the higher the flowrate, the faster it reaches the saturation point of the adsorbent. The breakpoints were 180, 260, and 420 minutes at 30, 20, 10 ml/min flowrate. The model data indicated that Thomas and Yoon-Nelson are fitted well with the experimental results. The models show the largest regression and the smallest error with the value of each 0.99 and 0.0035 at flowrate of 10 ml/min.

Keywords: *adsorption model; breakthrough curve; dye removal; fixed-bed column; flow rate*

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INTRODUCTION

Indonesia had various arts and cultures. One of the traditional arts is Jumputan fabric which produced by using tie and dye technic. In others part of Indonesia, Jumputan was known in various names. The name of Jumputan was known on Java and South Sumatera. Balinese people called it as Sangsangan, in Kalimantan known as Sasirangan, and Roto in Sulawesi. The Jumputan were mostly products of home industry that generally did not have good wastewater treatment. The Jumputan's wastewater contained a dye that was an organic compound. The wastewater discarded directly to the water could decrease levels of dissolved oxygen in the water.

A simple and economical technique to treat this wastewater is adsorption. It has been found that adsorption has a competitive result with other physical and chemical techniques, such as flocculation, coagulation, and precipitation, as they possess inherent limitations such as high cost, formation of hazardous by-products, and intensive energy requirements (Padmesh et al., 2006). Adsorption through fixed-bed columns has many advantages due to its simple and effective mode operation. It can reach high removal efficiency and be easily to scaled up from a laboratory to an industrial application (Ahmad and Hameed, 2010; de Franco et al., 2017). The adsorption of Jumputan's wastewater has several factors that influence its result, such as pH, pressure, flowrate, time, temperature, bed height, and concentration of the wastewater itself.

The activated carbon used in this study was produced from betel nuts. The highest component in the betel nuts was carbohydrate content of 60.86%, and the rest was water, protein, fat, and ash (Cundari et al., 2018a). Carbohydrates contained in the betel nuts forming a major component of the activated carbon in the amount of 86.27% based on the analysis of SEM-EDS (Cundari et al., 2018a).

Many researchers have conducted research on the study of adsorption on a batch and continuous system. The batch operation was preliminary research that reported the ability of the adsorbent and process, but not available in practice. The data of fixed-bed column operations should be used to design an industrial process (Hanbali et al., 2014). The time and shape of breakthrough curve were very important characteristics for evaluating the effectiveness of the adsorbent and the dynamic response behavior of a fixed-bed column (Busto Y. et al., 2016; Chu, 2020, 2004; Rosaria Augelletti et al., 2016). Measurement of isotherm and breakthrough curve based on experimental procedure can be time consuming and expensive (Poursaeidesfahani et al., 2019). Modeling of the transient adsorption process is one of the most efficient ways to select an appropriate adsorbent and find the optimal operating conditions (Poursaeidesfahani et al., 2019). The column adsorption models were tested with Thomas, Yoon-Nelson, and Adam-Bohart.

Hanbali et al. (2014) reported lead uptake using red algae on a batch and column experiments. The statistical analysis was used to identify the linear and nonlinear isotherms, kinetics, and models of adsorptioncolumn. Lim and Aris (2014) adsorbed Cadmium (II) and Lead (II) using calcareous skeleton (CS) in a continuous column. It evaluated by varying bed height, flowrate and adsorbate concentration. The breakthrough curve indicated that the highest bed height showed a maximum adsorption capacity of 26.447 and 38.460 mg/g for Cd (II) and Pb (II) respectively (Lim and Aris, 2014).

Darweesh and Ahmed (2017) utilized granular activated carbon to adsorb *Ciprofloxacin* (CIP) and *Norfloxacin* (NOR), then the data obtained were analyzed by Thomas, Adam-Bohart and Yoon-Nelson models. The result showed that the Adam-Bohart kinetic model has the lower values of R^2 (0.750-0.845) for CIP and (0.767-0.854) for NOR (Darweesh and Ahmed, 2017).

Recently, research on kinetics adsorption model of Jumputan's liquid waste onto betel nuts activated carbon has also been done in a batch process (Cundari et al., 2018b). The analysis result that the adsorption follows pseudo-second-order kinetic with R^2 of .929 and k value of 0.00008 $min^{-1}$and Langmuir model with R^2 of 0.999 and maximum adsorption capacity of 12.99 mg/g (Cundari et al., 2018b). Researches on Jumputan's liquid waste with continuous adsorption process have been conducted but only analyzed for COD (Cundari et al., 2017), TSS and BOD parameters. Therefore, this study is a continuation in the column adsorption model for Jumputan's liquid waste by a continuous process. The objective of this research was to find out the effect of flowrate of dye wastewater to the adsorption column performance onto betel nut activated carbon in a continuous fixed-bed column based on the breakthrough curve analysis.

MATERIALS AND METHODS

1.1 Adsorbent Preparation

Betel nut (*Cyrtostachys lakka*) used in this research taken from Universitas Sriwijaya, Indonesia. In adsorbent preparation section, the betel nut carbonized with the temperature of 500°C in a muffle furnace for 2 hours and activated by soaking the carbon with HCl 0.5 M for 1 hour. After that the carbon washed with aquadest until neutral and dried in an oven at 110°C for 3 hours. The betel nuts activated carbon was grinded to 60 mesh. The procedure of adsorbent preparation referred to Cundari et al. (2018a).

1.2 Adsorbate Preparation

The adsorbate used in this research was a synthetic solution. The procedure of making adsorbate and the material used in this stage is obtained directly from one of Jumputan craftsmen at Tuan Kentang Area, Palembang, Indonesia. The type of dye was direct synthetic dye. The synthetic dye used is dissolved into boiling water and the acetic acid. The initial concentration of synthetic dye used is 1 g/l and then add 15 ml of acetic acid to that solution.

1.3 Column Experiment

The fixed-bed adsorption column used is made from PVC with 2 inches of diameter and 60 cm of column length. The upper parts of this column contained a plastic sieve to distribute the adsorbate which placed 5 cm from the top. The adsorbent with a given amount was placed in the column with the depth of bed was 10 cm (100 g adsorbent). The adsorbent placed in 15 to 25 cm from the top of column. The activated carbon packed between cotton wool-plastic sieve layers to avoid loss of adsorbent. The adsorbate pumped from the top of column continuously. The effect of flowrate (10, 20, and 30 ml/min) on the breakthrough curve and dye removal was investigated. The effluent collected with time intervals until the saturation state (0, 20, 40, 60, 80, 120, 150, 180, 220, 260, 300, 360, 420). The influent and effluent samples analized using UV-Vis spectrophotometer at a maximum wavelength of 497 nm. The standard curve of the synthetic dye solution was y = 0.002x – 0.0439 with R^2 = 0.9938. The adsorption process is carried out under atmospheric conditions and room temperature. The schematic of the experimental set-up showed at figure 1.

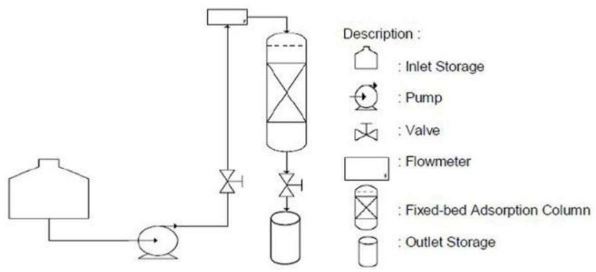


Figure 1: Schematic diagram of the experimental set-up

1.4 Mathematical Description of Adsorption Models

The experimental breakthrough curves analyzed with three common models represented by Thomas, Yoon-Nelson, and Adam Bohart. The Thomas model is one of the most commonly used models of column performance theory. The assumptions used in Thomas model are Langmuir isotherms, isothermal and isobaric operating conditions, no radial and/or axial dispersion, a constant column void fraction and second-order reversible kinetic adsorption (Lim and Aris, 2014; Talat et al., 2018; Zhang et al., 2011). The kinetic constant Thomas model (k_{Th}) and column adsorption capacity (q_{Th}) can be determined from the plot of $ln\left[\frac{C_0}{C_t}-1\right]$ to t at a given flowrate, respectively as slope and intercept (Sekhula et al., 2012; Talat et al., 2018). The Thomas model equation represented at (1), where C_o is the initial concentration of dye (mg/l), C_t is concentration of dye at time t (mg/l), m is the mass of adsorbent (g), and Q is the volumetric flow the feed (ml/min). The breakthrough equation for Thomas model represented at (2).

$$ln\left[\frac{C_o}{C_t}-1\right]=\frac{K_{Th}q_{Th}m}{Q}-K_{Th}C_o t \quad (1)$$

$$\frac{C_t}{C_o}=\frac{1}{1+\exp(\frac{k_{Th}\,q_{Th}\,m}{Q}-k_{Th}\,C_o\,t)} \quad (2)$$

The Yoon-Nelson model is a simple model by assuming that the rate of absorption decline for each adsorbate molecule is proportional to the probability of adsorption and breakthrough adsorption on the adsorbent (Talat et al., 2018; Zhang et al., 2011).The plot of the $ln\left(\frac{C_t}{C_0-C_t}\right)$ value of t will make a straight line with the slope showing the k_{YN} and intercept values indicating the $-\tau k_{YN}$ value. Based on the result, values, the column adsorption capacity of the Yoon-Nelson model (q_{YN}, mg/g). The Yoon-Nelson model equation showed at (3). The breakthrough equation for Yoon-Nelson model represented at (4).

$$ln\left(\frac{C_t}{C_0-C_t}\right)=k_{YN}\,t-\tau k_{YN} \quad (3)$$

$$\frac{C_t}{C_0}=\frac{\exp(k_{YN}\,t-\tau k_{YN})}{1+\exp(k_{YN}\,t-\tau k_{YN})} \quad (4)$$

The Adam-Bohart model assumes that the adsorption rate is equivalent with adsorption capacity and the adsorbed substance concentration. This model fit to describe the early part of the breakthrough curve (Hoces et al., 2010).The kinetic constant (k_{AB}) and column adsorption capacity (N_0) of this model can be determined from the plot of $ln\left(\frac{C_t}{C_o}\right)$to t, respectively as slope and intercept (Sekhula et al., 2012). The Adam-Bohart model equation described at (5). The breakthrough equation for Adam-Bohart model represented at (6).

$$ln\left(\frac{C_t}{C_o}\right)=\left(k_{AB}N_0\frac{Z}{F}\right)-k_{AB}C_o t \quad (5)$$

$$\frac{C_t}{C_o}=\exp\left(\left(k_{AB}N_0\frac{Z}{F}\right)-k_{AB}C_o t\right) \quad (6)$$

The kinetic model parameter is obtained by using nonlinear analysis according to quadratic of smallest error. In order to confirm which model is better to use, an error analysis (SS) is performed (Han et al., 2007). The relative mathematical formula of SS validated as follows (equation (7)), where $\frac{C_t}{C_o}exp$ is the data from experimental, $\frac{C_t}{C_o}cal$ is the data from calculation (by using Thomas, Yoon-Nelson, and Adam-Bohart models), and N

is the number of data that used to sketch breakthrough curve.

$$SS = \frac{\sum\left(\frac{C_t}{C_o}exp - \frac{C_t}{C_o}cal\right)^2}{N} \quad (7)$$

RESULTS AND DISCUSSION

In this continuous adsorption column, the variation of the synthetic dye solution flowrate used were 10, 20 and 30 ml/min respectively. Feed flowrate showed a negative effect on the saturation time, the higher the flowrate, the faster it reaches the saturation point of the adsorbent (de Franco et al., 2017; R. et al., 2018; Rout et al., 2017; Talat et al., 2018). It was due to the contact time between activated carbon and waste. The greater the flow rate, the contact time between activated carbon and waste will be faster so that breakthrough will also be achieved rapidly. That was the relationship between adsorbate and adsorbent at high flowrates that accelerate breakthrough and saturation (Busto Y. et al., 2016; de Franco et al., 2017; Erto et al., 2013; Garba A. et al., 2017; Han et al., 2007).

The experimental data of total dye absorption in a sequence was 47.6%; 56.9%; and 61.4%; for each flowrate of 30, 20, 10 ml/min. The time to reach breakpoints was 420, 260, and 180 minutes at flowrate 10, 20,30 ml/min respectively, as seen at Figure 2. The effectiveness of adsorption can be seen from the adsorption capacity (q_{exp}) from experimental procedure, with value of 25 mg/g for flow rate of 10 and 30 ml/min, and 29 mg/g for flow rate of 20 ml/min. By using this experimental data, parameters that describe at table 1 was obtained.

The mathematical modelling can help to analyze and explain experimental data, identify mechanisms relevant to the process, predict changes due to different operating conditions and to optimize the overall efficiency of the process (Borba et al., 2008). The prediction of a fixed bed column performance and also calculation kinetic constants and uptake capacities can be found by using mathematical breakthrough curve models (Crus-Olivares et al., 2013). Table 1 showed the kinetic adsorption of Thomas, Yoon-Nelson and Adam-Bohart parameters. The amount was calculated from the experimental data of dye removal concentration, with equation (1), (3), (5) and (7).

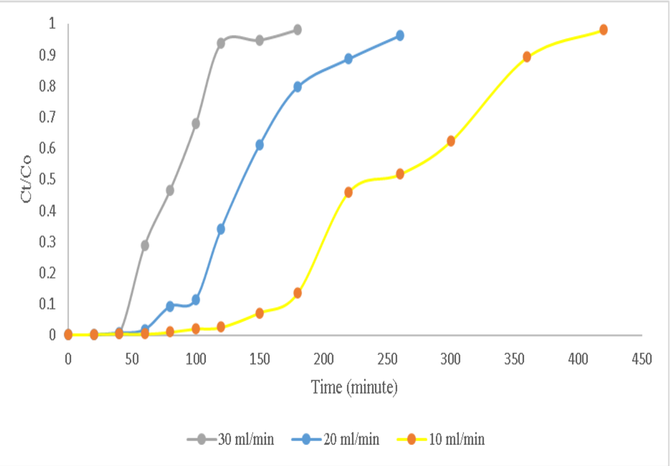


Figure 2: Experimental breakthrough curve of synthetic dye adsoption onto activated carbon

Table 1: Kinetic Adsorption Model Parameters

Kinetic Model	Parameter	Flowrate (ml/min) 10	20	30
Thomas Model	k_{Th}((l/(mg.min))	2.55 x 10^{-5}	4.13 x 10^{-5}	7.11 x 10^{-5}
	q_{Th}(mg/g)	25.30	30.27	28.94
	R^2	0.99	0.97	0.91
	SS	0.0035	0.0071	0.0185
Yoon-Nelson Model	k_{YN}(l/mg.min)	0.024	0.0393	0.0686
	τ (minute)	261.38	156.33	99.566
	q_{YN}(mg/g)	25.301	30.265	28.914
	R^2	0.99	0.9664	0.91
	SS	0.0035	0.0083	0.0205
Adam-Bohart Model	k_{AB}(l/mg.min)	0.0071	2.84 x 10^{-5}	4.607 x 10^{-5}
	N_o(mg/l)	0.0185	20135.356	20258.375
	q_{AB}(mg/g)	33.26	39.832	40.075
	R^2	0.91	0.84	0.75
	SS	0.6096	1.2478	3.5408

The Adsorption Column Models

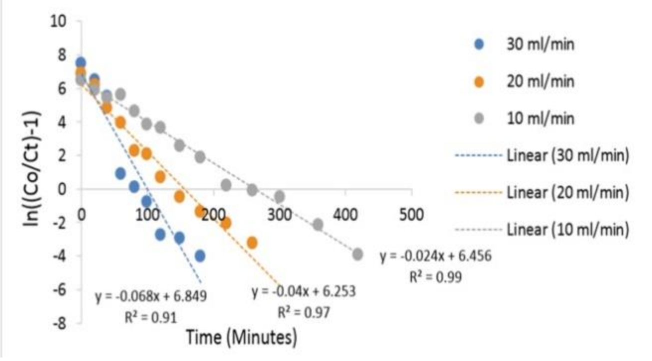
Thomas model was a model which commonly used to predict breakthrough curves from an adsorption column. This model can be used to evaluate the rate constant and maximum absorption of the adsorbate. Thomas's model helped in the study of the kinetics adsorption process of Jumputan's wastewater for each flowrate parameter. The results showed that each flowrate has a different coefficient of determination value. Each flowrate (10, 20 and 30 ml/min) has a regression coefficient (R^2) of 0.99, 0.97 and 0.91 respectively at Figure 3(a). With the allowed value of the fit to be used to describe the adsorption column kinetics happened (Sekhula, 2012). Kinetic constant (k_{Th}) and adsorption column capacity (q_{Th}) of Thomas Model at each flowrate showed in Table 1.

High flowrate will decreased q_{Th} and increased K_{Th}, this means driving forces for adsorption is concentration difference between dye on adsorbent and solution (Ahmad and Hameed, 2010; Talat et al., 2018). The q_{Th} calculated from Thomas model were very close with experimental data. The maximum adsorption capacity of Thomas Model (q_{TH}) achieved at 30.27 (mg/g) on 20 ml/min of flowrate.

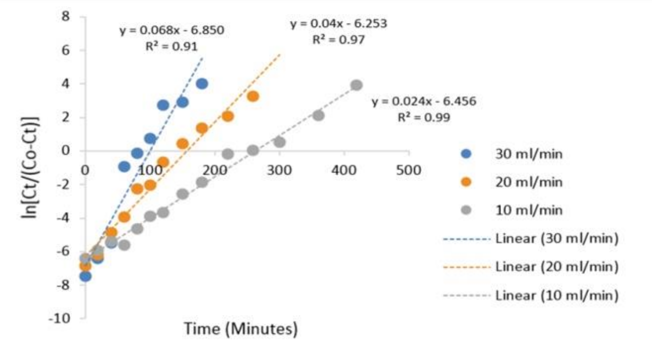
The Yoon-Nelson model is a relatively simple model assuming that the rate of decline in absorption for each adsorbate molecule was equivalent to the probability of adsorption and breakthrough adsorbate in adsorbent (Sekhula et al., 2012).This model is mathematically analogous to Thomas's model. This can be seen from the slope and intercept values obtained from the Thomas model plot and Yoon-Nelson model which is the opposite of Thomas's slope and intercept values. Regression coefficient value can be categorized in accordance with the value $R^2 > 0.90$ as in Figure 3(b).

At Yoon-Nelson kinetic constant and half-life, breakthrough was strongly influenced by the flowrate seen in Table 1. The increase of Yoon-Nelson kinetics constant occurred with increasing flowrate (Ahmad and Hameed, 2010). In addition, the half-life of the breakthrough would increase as the flowrate decreases. This is due to an increase in flowrate which will cause by rapid mass transfer as well so that the pores of activated carbon will be closed faster.

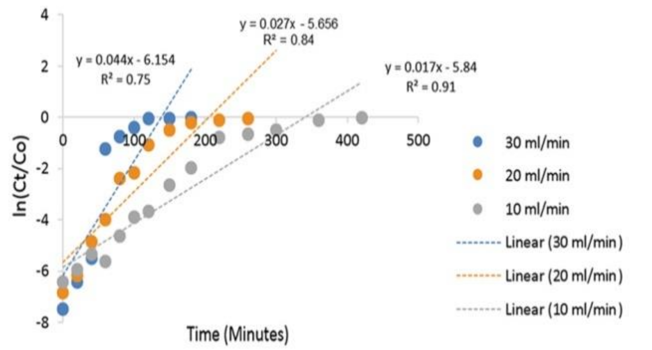
The Adam-Bohart model assumed that the adsorption rate was equivalent to the residual capacity of the solid and the concentration of the adsorbed agent. This model could be used to evaluate the adsorption rate and column adsorption capacity. The kinetic constants (k_{AB}) and adsorption capacity (q_{AB}) of the Adam-Bohart model at various flowrates showed in Table 1. The values of k_{AB} in Table 1 decreased with increasing flowrate (Ahmad and Hameed, 2010). An increase in the value of No (mg/l)



(a)



(b)



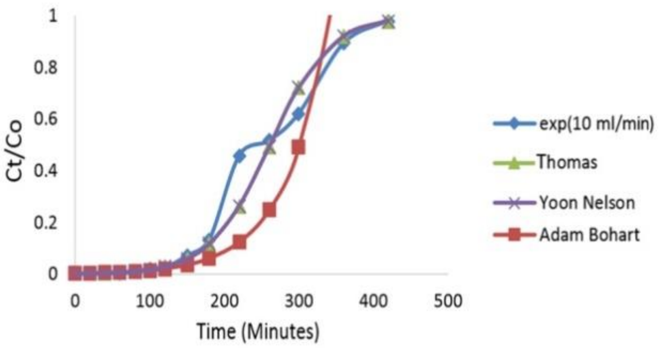
(c)

Figure 3: The Adsorption Column Models: (a) Thomas, (b) Yoon-Nelson, and (c) Adam-Bohart

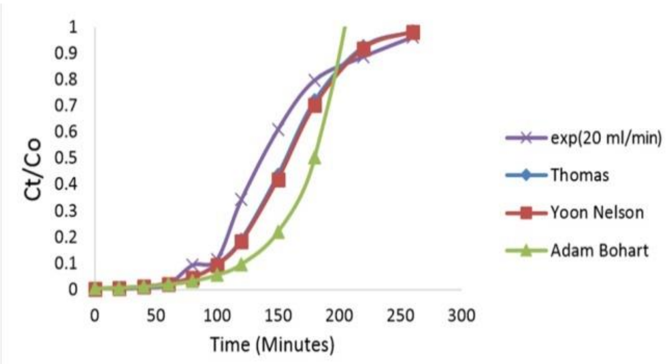
also occurred with an increase in flowrate values. On the Adam-Bohart kinetics plotted with R^2 value <0.90 as seen in Figure 3(c), It showed that suitability of this model with the data of research was low. According to that, the calculation of q_{AB} value according to Table 1 didn't match with the data of this research. The Adam-Bohart model was considered imprecise to describe the adsorption kinetics.

Breakthrough Curve Prediction

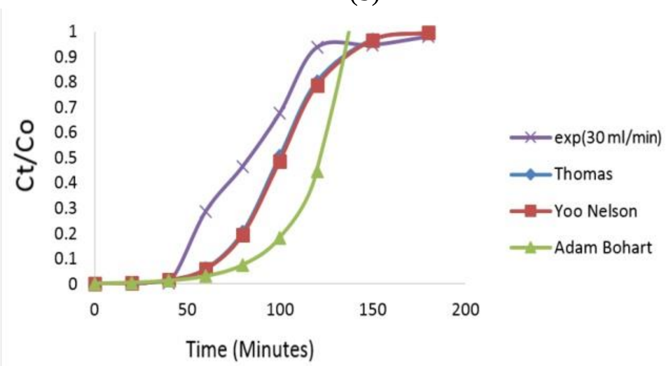
The breakthrough shape depend on individual transport process in column and in adsorbent (Ahmad and Hameed, 2010). Breakthrough is a very important characteristic to determine operation and dynamic response (Ahmad and Hameed, 2010). Sketching of a breakthrough curve by plotting $\frac{C_t}{C_o}$ versus t is showed at Figure 4. The experimental breakthrough curve



(a)



(b)



(c)

Figure 4: Predicted breakthrough curves: (a) flowrate of 10 ml/min, (b) flowrate of 20 ml/min, and (c) flowrate of 30 ml/min

shows that the curve is steeper at high flowrate than the lower one, that represented at Figure 2. The breakthrough curve for Thomas, Yoon-Nelson, and Adam-Bohart is calculated with equation (2), (4), and (6).

The parameter for determining the accuracy of a model was determined by the use of regression and the calculation of error analysis in the prediction of the breakthrough curve. In 1st Table, the largest regression and the smallest (SS) error were found at a flowrate of 10 ml/min. At the 10 ml/min of flowrate, the most accurate breakthrough curve estimated based on the three models were compared at the 20 and 30 ml/min flowrates seen in Figure 4 (a), (b), and (c). The model data indicated that Thomas and Yoon-Nelson are fitted well with the experimental results. The models show the largest regression and the smallest error with the value of each 0.99 and 0.0035 at 10 ml/min flowrate.

High flowrate will increase mass transfer and result faster saturation. As the effect of the faster saturation was lower adsorption capacity because insufficient time to contact and diffuse to the pores, solute left the column before equilibrium (Ahmad and Hameed, 2010). Figure 4 (a) and (b) showed that the prediction of the breakthrough curve in the Thomas and Yoon-Nelson models had a suitable form of breakthrough curve between experimental and kinetic model calculations, same result showed by (Ahmad and Hameed, 2010). It showed that Thomas and Yoon-Nelson models showed the best fit in adsorption kinetics. Based on the correlation value obtained in this study, the results showed that the three models had advantages and disadvantages of each. From the model of Thomas, Yoon-Nelson and Adam-Bohart, several earlier studies (Darweesh and Ahmed, 2017; Hanbali et al., 2014; Lim and Aris, 2014) suggested that the Thomas model had research data for the adsorption column matched with theoretical data so that it can be used as a reference for determining the mass, required for column operation under other operating conditions.

CONCLUSION

Increasing flowrate would decrease the removal of dye by the activated carbon. This statement fitted with the percentage data of total dye adsorption in a sequence of 47.6%; 56.9%; and 61.4%; for each flowrate of 30, 20, 10 ml/min. Feed flow rate showed a negative effect on the saturation time, the higher the flow rate, the faster it reaches the saturation point of the adsorbent. It was supported by the faster of time to reach breakpoints of 180, 260, and 420 minutes at 30, 20, 10 ml/min flowrate. The Thomas and Yoon-Nelson kinetic models are fitted well with the experimental results that give the greatest regression and the smallest error analysis, with value 0.99 and 0.0035 at flowrate of 10 ml/min.

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