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Recovery of Minor Palm Oil Compounds Using Packed Bed Adsorption Column

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Abstract

Carotene and tocopherol are valuable products that exist as minor compounds in palm oil and mostly extracted out during many stages of palm oil processing. Hence, most of it ended up in wastewater or palm oil mill effluent (POME). Fortunately, adsorption is potentially one of the most efficient method as compared to the others. In fact, it is widely studied in laboratory scale, in order to obtain equilibrium data for the steady state system. However, industrial practices are mostly operated in unsteady state in a continuous manner. Consequently, this study is executed to design a recovery process of one of the minor compounds in palm oil mill effluent (POME), which is carotene, using silica gel. It aims to predict the dynamic adsorption of recovery of minor compounds from palm oil mill effluent based on available equilibrium data, investigate the effects of dynamic and physical properties of the system towards the process by analyzing the breakthrough curve and study the feasibility of the scale up process by performing a sensitivity analysis on the system. Then, a base simulation was prepared by using available equilibrium data. Operating and design parameters such as, bed height, inlet flowrate and concentration were manipulated. Consistent with previous packed column studies, increase flow and concentration will reduce the time required for the column to achieve saturation, while increase bed height effects were vice versa. Finally, the last objective to achieve was to study the practicality of the packed bed column and perform a sensitivity on assumptions and predictions such as predicted mass transfer coefficient and isotherm model. It is proven that the selection of isotherm model and prediction in coefficient did not pose a large impact to the breakthrough curve and the average time required for the column of 1.5 m tall and 0.8 in diameter, to reach breakthrough time is 1.7 days. Hence, it can be concluded that adsorption technology using silica gel as its adsorbent can be applied is recovering minor compounds in palm oil mills.

INTRODUCTION

Malaysia is known to be one of the major producer of palm oil in the world, with an estimated value of 8.3 and 16.3 million tonnes in 2000 and 2008 (Foo & Hameed, 2009). There is a total of 423 mills in Malaysia in both years that managed to produce 89 million tonnes of fresh fruit bunches every year. Hence, resulting a mounting value of 66.8 million tonnes of POME discharged (Vairappan & Yen, 2008).

Palm oil mill effluent (POME) is the wastewater or effluent produced during palm oil production. It is a viscous acidic brown liquid

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(Sangkharak et al., 2016). It is mainly organic and non – toxic in nature. However, it emits a foul odor. Malaysia is among the largest POME producers, with production of 8.3 and 16.3 million in 2000 and 2008, respectively (Salihu & Alam, 2012). The value was proven to be increasing with each passing year as demand for oleo – based products keep growing exponentially (Ibrahim et al., 2012). Generally, crude palm oil (CPO) is one of the world's largest resource of carotenes. It contained 1% minor components, including carotenoid, tocopherols and sterols (Madaki & Seng, 2013). The concentration of the minor components usually ranges between 400 and 3500 ppm, with 15 times more than carrots (Ahmad et al., 2008). Consequently, most of the nutrients were extracted out of the oil and into the wastewater during washing and polishing.

POME also requires expensive treatment as is has a high Biochemical Oxygen Demand (BOD) (Igwe & Onyegbado, 2007; Kamin et al., 2020). To further understand the characteristics of POME, the following Table 1 is a list of the average values of the parameter of POME.

Table 1. Chemical Constituents in Palm Oil Mill Effluent (Salihu & Alam, 2012).

Constituents	Compositions (%)
Moisture	6.99 ± 0.14
Crude Protein	12.75 ± 1.3
Crude Lipid	10.21 ± 1.24
Ash	14.88 ± 1.35
Carbohydrate	29.55 ± 2.44
Nitrogen Free Extract	26.39 ± 2.33
Total Carotene	0.019 ± 0.001

Many methods such as distillation column, membrane filtration and adsorption that can be used to recover minor compounds from POME. However, adsorption is proven to be the most economical as it requires minimal energy and simple operational condition (Boudrahem et al., 2011; Tien, 2019).

In this research, one of the applications of adsorption was studied, which is the recovery of valuable compounds in minute concentration from bulk fluid. The minor compounds that needed to be recovered are large biomolecules of vitamin precursors, such as carotene and tocopherol, from palm oil mill effluent.

In this study, the dynamic adsorption process was simulated using Aspen Adsorption V7.3. Its objectives are to find the breakthrough curve of adsorption process of carotene from palm oil mill effluent and the factors that affects the breakthrough time. Then, the process undergoes scaling – up to suit industrial release of POME and sensitivity analysis is also performed to evaluate the sensitivity of the predicted parameters towards the breakthrough time.

MATERIALS AND METHODOLOGY

This study is based on data from a research article published in Chemical Engineering Journal, by Ahmad et al. (2009) on "Adsorption kinetics and thermodynamics of β-carotene on silica-based adsorbent". This article is taken as reference for simulation purpose. The dynamic simulation of the packed bed adsorption column is run using the Aspen Adsorption V7.3 simulation tool as used by other authors (Anisuzzaman et al., 2016; Bono et al., 2007; Kamin et al, 2017).

The required data of adsorbent particle properties and isotherm equilibrium is extracted directly from the article, as stated in Table 2 and 3. The unknown parameter of mass transfer coefficient and molecular diffusivity are estimated using widely known correlation in the literature.

Table 2. General Properties of Silica Gel

Silica Gel Properties	Description
Form	Bead / granular
Mean pore radius (nm)	1 to 12 nm
BET surface area (m^2/g)	250 - 900 m^2/g
Bulk density (kg/m^3)	720.83
Particle density	0.7 – 1.0 g/cm^3
Total porosity	0.5 – 0.65
Pore volume	0.45 – 1.0 cm^3/g

Table 3. Isotherm parameters for carotene onto silica gels (Ahmad et al., 2009)

Langmuir isotherm @ 30°C	K_L (L/mg)	Q_m (mg/g)
	0.0089	22.422
Freundlich isotherm @ 30°C	K_F [(mg/g)/(mg/L)^n]	n (-)
	0.8204	0.5408

Simulation

This section provides the assumptions used is a dynamic simulation of a packed bed adsorption column in Aspen Adsorption V7.3 simulation tool.

(a) Discretization Method: Upwind Discretization Scheme 1 (UDS1)
(b) Momentum Balance: Convection only
(c) Pressure Drop Assumption: None
(d) Velocity Assumption: None
(e) Kinetic Model: Linear Lumped Resistance
(f) Film Model: Solid
(g) Isotherm: Langmuir 1 or Freundlich 1
(h) Energy Balance: Isotherm

Continuity Equation

The packed bed adsorption process could be modelled by ideal plug flow model as it is sufficient to represent the flow in the packed bed system (Xu et al., 2013). The dispersion term is negligible, which leads to momentum balance of convection only. The mathematical model is shown as in Eq. (1) (Farhadpour & Bono, 1996)

$$\varepsilon \frac{\partial C}{\partial t} + \upsilon \frac{\partial C}{\partial z} + (1-\varepsilon)\rho_a \frac{\partial q}{\partial t} = 0 \quad (1)$$

where ρ_s is the bulk adsorbent density (kg/m^3), υ is the interstitial velocity (m/s), z is the bed axial position (m), t is the process time (s), c is the aqueous-phase concentration of carotene component (mg/L) and q is the solid-phase loading of carotene component (mg/g).

Kinetic Model

The assumption of linear driving force (LDF) approximation is used as a kinetic model in this dynamic simulation study. The LDF approximation is expressed as in Eq. (2) (AspenONE, 2009).

$$\frac{\partial \overline{w}}{\partial t} = MTC(w^* - w) \quad (2)$$

where q* is solid-phase loading of the carotene component at the interface and MTC is the LDF mass transfer coefficient (1/s). The LDF mass transfer coefficient can be calculated combining the coefficient of external mass transfer resistance and intraparticle surface resistance as in Eq. (3) (Poursaeidesfahani et al., 2018; Shafeeyan et al., 2014; Siahpoosh et al., 2009).

$$\frac{1}{MTC} = \frac{R_p}{3k_f} + \frac{{R_p}^2}{15D_s} \quad (3)$$

where R_p is the adsorbent particle radius (m).

Mass Transport Estimation

The kinetic parameters such as external film mass transfer diffusion k_f, aqueous-phase diffusivity D_m, and intraparticle mass transfer diffusion k_s were estimated using well-established correlations from literature. The aqueous-phase diffusivity D_m was estimated using correlation suggested as in Eq. (4) (Worch, 2008).

$$D_L = \frac{3.595 \times 10^{-14} T}{\eta M^{0.53}} \quad (4)$$

The external film mass transfer diffusion k_f is calculated using Sherwood correlation and several dimensionless number. For the range of $0.001 < Re < 5.8$ found in this work, the governing equation used for the Sherwood correlation to calculate the film mass transfer diffusion, k_f (m/s) is shown in Eq. (5) (Ohashi et al., 1981).

$$Sh = 2.0 + 1.58 Sc^{0.33} Re^{0.4}; \quad 0.001 < Re < 5.8 \quad (5)$$

The intraparticle surface diffusion, D_s (m^2/s) is calculated using empirical correlation based on work of Worch (2008) was used as presented in Eq. (6) (Glueckauf, 1955; Worch, 2008).

$$D_s = 8.6 \times 10^{-5} R_p \sqrt{\frac{D_L c_0}{w_0}} \quad (6)$$

where c_0 is aqueous-phase inlet concentration of carotene component (mg/L) and q_0 is the corresponding solid-phase loading of the carotene component at c_0 (mg/g).

Industrial – Scaled Packed Bed Column Dimensions

The carotene adsorption onto silica gels adsorbent in packed bed column was scaled-up to obtain a column operational time within two days. The parameters value of an industrial scaled bed column is presented in Table 4.

RESULTS AND DISCUSSION

Base Simulation Breakthrough Curve Establishment

Base simulation needed to be established as a reference point, prior from studying the effecting parameters. Table 5 is the initial values of

Table 4. Specification for Industrial Scale Packed Bed Column. (Ahmad et al. (2009), Salihu and Alam (2012)**).**

Parameter	Value
Inlet flowrate, F (m^3/s)	1.00×10^{-4}
Inlet concentration, C (mg/L)	190
Column height, H (m)	1.50
Column diameter, D (m)	0.80
Bed porosity, ε (-)	0.65
Bulk adsorbent density, ρ_s (kg/m3)	720
External film mass transfer diffusion, k_f(m/s)	2.13×10^{-5}
Intraparticle (surface) mass transfer diffusion, k_s (m^2/s)	3.8126×10^{-13}
Langmuir isotherm	
Q_m (mg/g)	22.422
K_L (L/mg)	0.0089
Freundlich isotherm	
K_F [(mg/g)/(mg/L)^n]	0.8204
n (-)	0.5408

Table 5. Model parameters value for base case simulation.

Parameter	Value
Inlet flowrate, F (mL/min)	12.0
Inlet concentration, C (mg/L)	50
Column height, H (m)	0.80
Column diameter, D (m)	0.01
Bed porosity, ε (-)	0.65
Bulk adsorbent density, ρ_s (kg/m3)	720
External film mass transfer diffusion, k_f(m/s)	5.01×10^{-5}
Intraparticle (surface) mass transfer diffusion, k_s (m^2/s)	2.7938×10^{-13}
Langmuir isotherm	
Q_m (mg/g)	22.422
K_L (L/mg)	0.0089

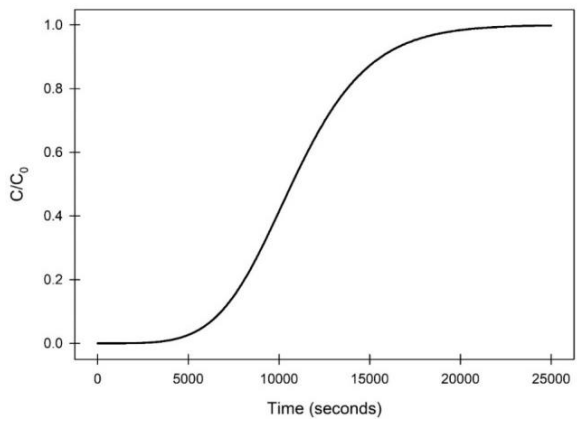


Figure 1. Base case simulation breakthrough curve.

specifications. The values will be the same throughout the study except the manipulated variable.

The plot of the breakthrough curve for base case simulation is shown in Figure 1. From the figure, it indicates that the system achieved breakthrough time at 5792 sec with the bed operational condition as stated in Table 5. The breakthrough curve is consistent with other researchers from literature in terms of its similar "S" shaped curve (Anisuzzaman et al., 2016; Sperlich et al., 2008; Vera et al., 2019).

Factors Influencing Column Performance

There are several factors that may affect the breakthrough curve, which are the inlet carotene concentration, inlet carotene flowrate and bed column height. The simulation was repeated several times with the base simulation as reference, at ±20% from base simulation parameters value. The different between the repeated simulations were the value of the parameters and its effect on the breakthrough curve was studied.

Inlet flowrate

The inlet flowrate also affects the breakthrough curve of the adsorption column. The flowrates tested were 9.6, 12 and 14.4 mL/min. Other parameters such as inlet concentration and bed column height were kept constant at base simulation values.

The plot of the effect of the inlet flowrate was presented in Figure 2(a). When the inlet flowrate was increased from 9.6 to 14.4 mL/min, the breakthrough time decreases from 8050 to 4350 sec. This is because the residence time is insufficient to achieve equilibrium at higher flowrate (Babu & Gupta, 2005). It shows that the mass transfer zone of the adsorptions became smaller as well. This may be due to the rate of available adsorbate passing through the adsorbent. Silica gel adsorbent starts to saturate faster due to the high rate of available adsorbate passing through. Higher inlet flowrate decreases the contact time between adsorbate and adsorbent and leads to a decrease in adsorption capacity and service time of the bed column Hymavathi & Prabhakar, 2019).

Inlet concentration

Theoretically, higher concentration indicates a higher amount of carotene compound in the solution. From the base simulation, the

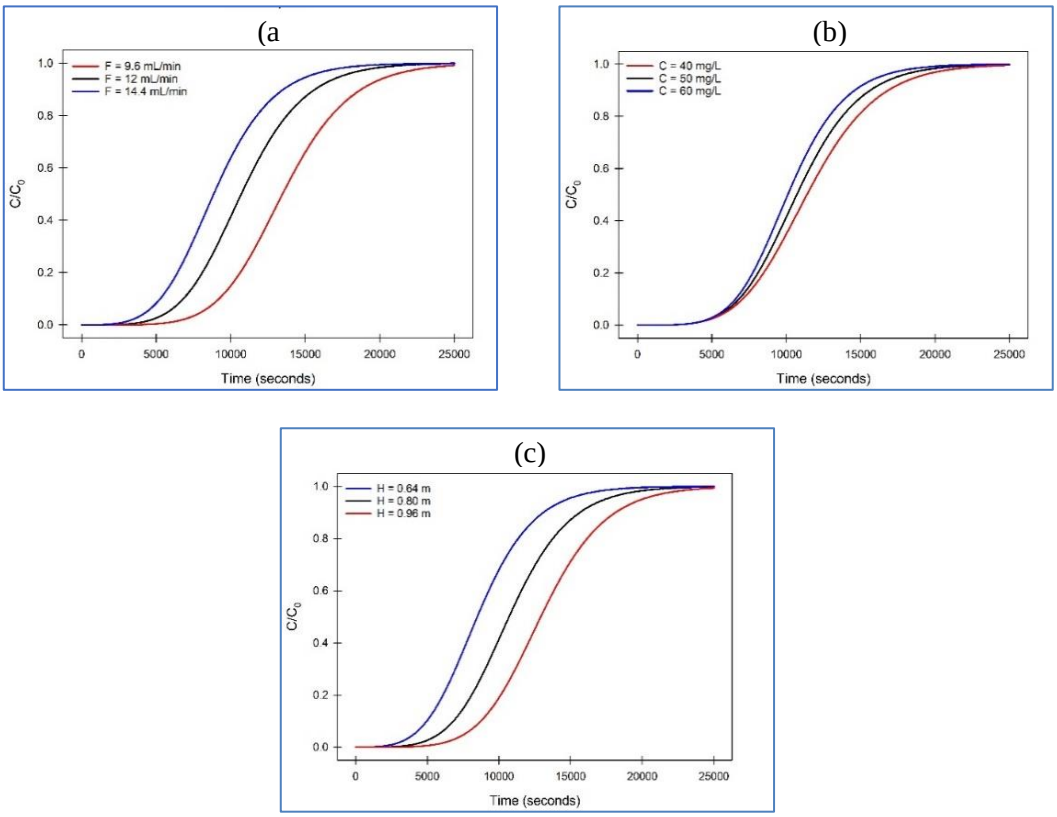


Figure 2. Effect of (a) inlet flowrate [C=50 mg/L, H=0.8 m, D=0.01 m]; (b) inlet concentration [F=12 mL/min, H=0.8 m, D=0.01 m]; (c) bed column height [F=12 mL/min, C=50 mg/L, D=0.01 m] on the breakthrough curve.

effect of inlet concentrations of ±20% from the base simulation value were studied, which were 40, 50 and 60 mg/L.

The results obtained as shown in Figure 2(b) shows a reduction in breakthrough time with increasing initial concentration. When the concentration is increased from 40 to 60 mg/L, the breakthrough time of the bed column decreases from 5942 to 5640 sec. This may due to the increased amount of available carotene in the solution that increases the breakthrough time of silica gel adsorbent. Hence, silica gel will have to adsorb more carotene from the solution, resulting a faster exhaustion rate for the bed column and increased the adsorption capacity of the bed (Levan et al., 2009)

Column height

Another parameter that was studied was bed column height, which is the height of the adsorbent in the packed bed column. The bed column height investigated were 0.64 m, 0.80 m and 0.96 m, while other parameters were kept constant such as inlet flowrate and inlet concentration. The plot of effect of bed column height is shown in Figure 2(c).

From the figure, it can be observed that the breakthrough time is directly proportional to the bed column height. The breakthrough time of the system increases from 4065 to 7590 sec when the bed height is increased from 0.64 to 0.96 m. The length where most of the adsorbates are removed are called the adsorption zone and it is arbitrary depending on the amount of solutes in the bulk fluid (Gabelman, 2017). As the fluid traverses in the column, it will be further adsorb and the column is slowly being saturated with the solute (Poursaeidesfahani et al., 2019). Hence, increasing the adsorption zone like a slow wave. With increased bed column height, the adsorption zone can be much larger and reaches the breakthrough time longer as seen in Figure 2(c) (Kulkarni, 2017).

Industrial – Scaled Dimensions And Sensitivity Analysis

The breakthrough curve of the up-scaled system is shown in Figure 3. From the breakthrough curve, it shows that the system achieved breakthrough time in 1.7 days.

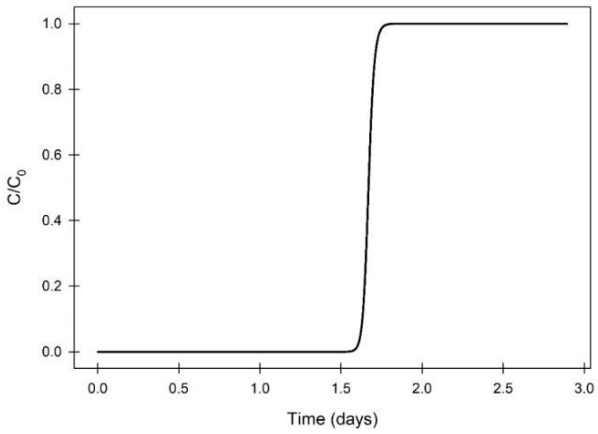
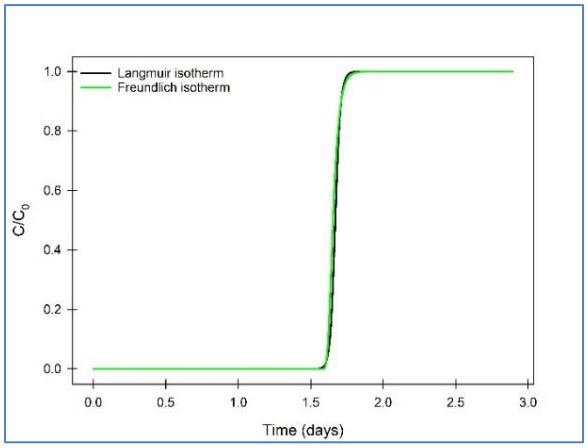


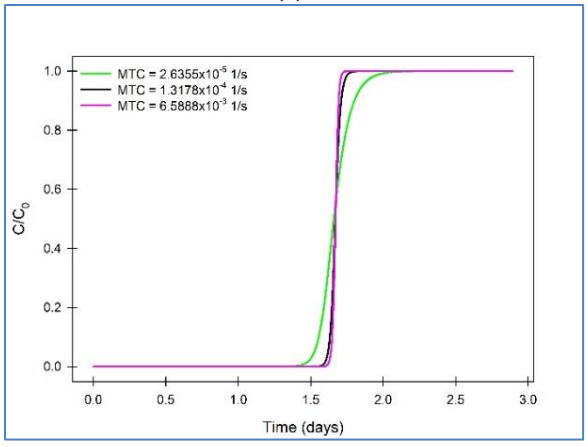
Figure 3. Up-scaled breakthrough curve

Adsorption isotherm model

The selection of the isotherm model of the breakthrough curve of up-scaled system was studied. The results were plotted as shown in Figure 4 (a).



(a)



(b)

Figure 4. Sensitivity analysis of (a) isotherm model; (b) mass transfer coefficient on the system's performance. The parameters value of inlet flowrate, inlet concentration, bed height and bed diameter are presented in Table 4.

From Figure 4(a), it shows that the effect of isotherm model is not significant for Langmuir model and Freundlich model. This can be predicted from the correlation coefficient that both models produced from the adsorption isotherm investigation in batch experimental work as done by Ahmad et al. (2009), with R^2 of 0.9813 and 0.9568 for Langmuir and Freundlich model, respectively. However, in this case the Langmuir model is much more preferable than Freundlich model because the R^2 is much higher and nearer to unity.

Mass transfer coefficient

Generally, mass transfer coefficient determines the slope of the breakthrough curve. Larger MTC value leads to a steeper curve, while smaller value results in a less steep curve. Since MTC values were calculated using correlations, it is perfectly suitable to be included in the sensitivity analysis study. The deviation in MTC studied are within the range as stated in Table 6.

Table 6. Changes range of mass transfer coefficient investigation.

Range	**Mass transfer coefficient, MTC (1/s)**
Five-fold decrease	2.6355×10^{-5}
Original value	1.3178×10^{-4}
Five-fold increase	6.5888×10^{-4}

The breakthrough curve of the bed adsorption system is not greatly affected by the deviation in mass transfer coefficient. From the Figure 4(b), it is obvious that the lower value of MTC by five-folds decreases from the original value is more significant than the five-folds increase in the MTC. The slow approach of C/C_0 towards 1 was observed at lower MTC due to slow intraparticle surface diffusion within the pores of silica gels adsorbents (Bono, 1989; Lin et al., 2017).

CONCLUSION

This study aimed to predict the dynamic behaviour for adsorption uptake of carotene onto silica gels in a packed bed column. With appropriate required input data, the dynamic behaviour of silica gels packed bed column for carotene uptake can be predicted using Aspen Adsorption V7.3. The effect of inlet concentration, inlet flowrate and column height towards the

dynamic behaviour of the system were investigated by manipulating one variable at a time, at ±20% around the base simulation value, while keeping other variable fixed. Then, the silica gels packed bed column was scaled up for industrial application in recovering large amount of carotene from POME discharge. A sensitivity analysis was conducted on the isotherm model selection and mass transfer coefficient to study how much deviation resulting from wrongly selected or predicted parameters.

Based on this present study, some conclusions could be drawn. First, the results can be separated into two, which are the parameters the reduced the breakthrough time, such as inlet concentration and inlet flowrate, and the parameters that increased the breakthrough time, like bed height. The findings from this section were applied when setting up the industrial scale simulation in order for it to achieve breakthrough within the desired time. The second part is scaling up the process so it fits with the industrial discharge of POME. In general, the discharge of POME is pretty large and can get up 21,000 kg every day. Meanwhile, the recovery of minor compound must be done within two to three days in order for it to be profitable. Hence, the parameters of the column and flow must be adjusted so that the silica gel reached it saturated point in the second or third day, then it will be regenerated. In order for the process to be done in two days, the bed height must 1.5 m tall with a 0.8 m column diameter. The flow was set to be 1.00×10^{-4} m^3/s and the initial concentration is 190 ppm. In addition, the sensitivity analysis revealed that, for the case at hand, the isotherm model and mass transfer coefficient are not greatly affecting the system's performance.

Overall, this study partially filled the gap in the adsorption study to expand the investigation on continuous, unsteady state adsorption process. All in all, adsorption process is applicable for industrial application in turning waste into resource by recovering valuable minor compounds in one of the largest waste product in Malaysia.

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SYMBOLS

Re	= Reynolds number	[-]
Sc	= Schmidt number	[-]
Sh	= Sherwood number	[-]
k_f	= External film diffusion	[m/s]
D_s	= Intraparticle surface diffusion	[m^2/s]
R_p	= Particle radius	[m]
c_o	= Inlet concentration	[mg/L]
q_o	= Amount adsorbed on solid phase at c_o	[mg/g]
q*	= Amount adsorbed on solid phase at interface	[mg/g]
K_F	= Freundlich parameter 1	$\left[\frac{(mg/g)}{(mg/L)^n}\right]$
n	= Freundlich parameter 2	[-]
Q_m	= Langmuir parameter 1	[mg/g]
K_L	= Langmuir parameter 2	[L/mg]
D_L	= Aqueous-phase diffusivity	[m^2/s]
T	= Temperature	[K]
M	= Molecular weight of solute	[kg/kmol]

Greek Letters

ε	= Bed porosity	[m^3 void/m^3 bed]
ρ_s	= Bulk solid density	[kg/m^3]
v_F	= Fluid interstitial velocity	[m/s]
ν	= Kinematic viscosity of solvent	[m^2/s]
η	= Dynamic viscosity of solvent	[Pa.s]

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