



Removal of COD, TSS and colour from palm oil mill effluent (POME) using montmorillonite

Muhammad Said^{a,c}, Hassimi Abu Hasan^a, Mohd Tusirin Mohd Nor^a,
Abdul Wahab Mohammad^{a,b,*}

^aFaculty of Engineering and Built Environment, Department of Chemical and Process Engineering, Universiti Kebangsaan Malaysia, 43600 UKM Bangi, Selangor Darul Ehsan, Malaysia, Tel. +60102637864; email: msaidpalembang@yahoo.co.id (M. Said), Tel. +60102400530; email: simiabuhasan@gmail.com (H.A. Hasan), Tel. +603 8921 6103; email: tusirin@eng.ukm.my (M.T. Mohd Nor), Tel. +603 8921 6410; Fax: +603 8921 6148; emails: wahabm@eng.ukm.my, drawm67@gmail.com (A.W. Mohammad)

^bFaculty of Engineering and Built Environment, Research Centre for Sustainable Process Technology (CESPRO), Universiti Kebangsaan Malaysia, 43600 UKM Bangi, Selangor Darul Ehsan, Malaysia

^cFaculty of Mathematics and Science, Department of Chemistry, University of Sriwijaya, Indralaya 30662, Sumatera Selatan, Indonesia

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ABSTRACT

The adsorption of POME using natural clay, montmorillonite, has been investigated. The reductions of three important parameters (chemical oxygen demand, total suspended solids and colour) in various influencing factors have been determined. The optimum conditions for mixing speed of the stirrer, contact time, dosage of adsorbent, concentration of palm oil mill effluent (POME) and pH were 300 rpm, 90 min, 5 g/L, 25% POME and pH 7, respectively. The removal reached more than 95% for all parameters. The experimental data were found to fit with the Langmuir adsorption isotherm model. The intraparticle diffusion model controlled the adsorption process and, therefore, the diffusion of the solute into the inside pores of the adsorbent took places permanently (irreversible) because the particles were trapped inside the pores. This situation causes the adsorbent capacity to decrease and it cannot be used continuously prior to the regeneration or activation, both chemically and thermally.

Keywords: POME; Montmorillonite; Adsorption; Langmuir model; Intraparticle diffusion

1. Introduction

Malaysia and Indonesia have been known as the world's leading producers of palm oil. Along with increasing production of palm oil, the need for clean water also increases. To process 1 ton of fresh fruit bunches, 5–7.5 tons of fresh water is required; unfortunately, 50% of this water will end up as a palm oil

mill effluent (POME), whereas the rest is lost through steam boilers and leakages. These wastewaters will potentially pollute the environment if they do not receive proper handling [1].

POME contains 95–96% water, 0.6–0.7% oil and 4–5% total solids. It is acidic (pH 4–5), dark in colour and foul smelling. In addition, POME has a high organic content (chemical oxygen demand (COD) 50,000 mg/L and BOD 25,000 mg/L). Based on these

*Corresponding author.

characteristics, an efficient and inexpensive method is required for treating POME.

Numerous techniques have been used to treat POME, such as ultrafiltration [2], the Fenton process [3], an EGSB reactor [4], fixed-film bioreactors [5] and adsorption [6]. Among the technologies, adsorption is one of the most commonly used methods to remove pollutants from a solution.

Ahmad [7] has conducted research using chitosan powder and flakes. The results showed that the adsorption performance of chitosan powder is better than the flakes of chitosan. To adsorb 99% of residue oil, 0.5 and 2 g/L of chitosan powder and flakes were needed, respectively, where the initial residue oil content in POME was ± 2.0 g/L. In addition, there is no big difference in residue oil adsorption between the chitosan powder and the flakes at various pH values of POME. Moreover, rubber powder has also been investigated as an adsorbent to adsorb residual oil from POME. It was found that about 30 g of the synthetic rubber powder was needed to remove 88% of 850 mg/L residual oil. The increasing mixing speed and mixing time caused an increase in oil removal [8].

Zeolites are natural adsorbents, which have been applied for the removal of heavy metal ions (zinc, manganese and iron) from POME. Shavandi [9] investigated the adsorption capacity of natural zeolites by increasing the pH values of POME. It was found that heavy metal ion removal raised the pH, and pH 7 was considered as the optimum. On the other hand, a dosage of 25-g adsorbent for 250 mL of POME was determined as the optimum dosage and, by accelerating the stirring rate, it is also enhances the sorption capacity of the zeolite.

Most previously reported studies on adsorption processes using clay as the adsorbent discuss the removal of metals [10–15]. Although activated carbon is still the most popular adsorbent, the expensive cost and difficulties in regenerating the material are forcing researchers to search for a new adsorbent.

Montmorillonite is a natural adsorbent that is commonly used. This is not only because of its large surface area, adsorption and ion-exchange capacity, but also its chemical and mechanical stability. Montmorillonite has two tetrahedral layers that sandwich one octahedral layer. The tetrahedral layers of montmorillonite contain Si^{4+} as the centre atom, whereas Al^{3+} is central in the octahedral layer. The absorption capabilities come from its negative charge. This negative charge from montmorillonite neutralises the positive charge of impurities and then attracts and traps them on the surface of the adsorbent [16].

Adsorption of the herbicide paraquat by clay mineral has been investigated by Tsai and Lai [17]. The rapid adsorption of the pollutant is based on the ion-exchange process from clay mineral and paraquat. The adsorption capacity increased as the initial pH decreased, but the opposite occurred for the concentration of salinity. The order of affinity of ions has the same result as it does with temperature. Increasing the affinity and temperature makes the adsorption capacity increases.

Tsai [18] studied the kinetic parameters of a basic dye adsorbed by regenerated and spent bleaching earth. The result shows a second-order reaction model, which can describe the adsorption process, but the fit is not so good for the intraparticle diffusion model. In addition, the adsorption process is dependent on both the pH and temperature of solution. pH values of 3 and 11 as well as increasing temperature from 15 to 45°C are the best conditions to reach the maximum adsorption capacity.

POME has a high organic content, and these compounds are difficult to remove using conventional treatment. Studies of the adsorption processes are needed with the aim to eliminate the organic content, which is characterised by a reduction in the COD value. The objective of this research was to determine optimised conditions when using montmorillonite as the adsorbent in reducing variation parameters of POME, such as COD, colour and total suspended solids (TSS). The effects of the mixing speed of stirrer, contact time, adsorbent dosage, concentration of POME and pH were investigated. Both adsorption isotherm models were applied to calculate the constant rate and adsorption capacity. In addition, the pseudo-first- and -second-order models were adopted to determine the mechanism of adsorption onto montmorillonite. The final discharge pond effluent from a palm oil mill was used as the sample.

2. Materials and methods

2.1. Characterisation of montmorillonite and POME

Montmorillonite was purchased from the Acros Organics and used as received. The data of the physical and chemical compositions were provided by the supplier and are listed in Table 1.

The POME sample was collected from an aerobic pond of West Palm Oil Mill in Sime Darby Sdn. Bhd., Carey Island, Malaysia. The POME was collected in a container and preserved at 4°C. The characteristics of the POME are summarised in Table 2.

Table 1
Physical and chemical properties of montmorillonite

Physical properties	Value
Colour	Beige to grey
Surface area (m ² /g)	240
Bulk density (g/L)	370
Micro pore volume (ml/g):	
0–80 nm	0.36
0–24 nm	0.30
0–14 nm	0.26
Chemical composition (%):	
SiO ₂	73.0
Al ₂ O ₃	14.0
Fe ₂ O ₃	2.7
CaO	0.2
MgO	1.1
Na ₂ O	0.6
K ₂ O	1.9

Table 2
Characteristics of POME in the final pond of a local palm oil mill factory

Parameter	Feed sample
COD (mg/L)	12,040
TSS (mg/L)	3,103
Colour (Pt Co)	54,200
pH	7.43

2.2. Experiment set-up

The experiments were performed in 500-mL beakers containing 250 mL of POME solution. The variable conditions were the type of adsorbent (activated carbon or montmorillonite), dosage of adsorbent (2, 3, 4 and 5 g/L), pH (3, 5, 7 and 9), contact time (30, 60, 90 and 120 min), mixing speed (300 and 500 rpm) and concentration of POME solution (100, 75, 50 and 25%).

Each adsorption process was the combination of different variable conditions. After the adsorption was completed, the solution was left for 1 h to make the adsorbent settle at the bottom, and then the clear solution was transferred to a new container for analysis.

The characterisation of the feed and permeate samples was performed to analyse the COD, TSS, colour and turbidity, using a portable data logging HACH spectrophotometer DR 3900 (USA). The experiments were repeated three times and the average values were recorded. Reductions in the parameters of the solution were calculated by the following equation:

$$C (\%) = \left(1 - \frac{C_p}{C_s}\right) \times 100 \quad (1)$$

where C_p is the concentration in the permeate solution and C_s is the concentration of the feed sample.

The equilibrium concentration (Q_e) after adsorption using montmorillonite can be determined by the formula:

$$Q_e = \left(\frac{C_s - C_p}{m}\right) \times V \quad (2)$$

where m is the mass of adsorbent and V is the volume of solution.

3. Results and discussion

3.1. Type of adsorbent (pH 7, 60 min and 300 rpm)

The removal efficiency of montmorillonite for COD, TSS and colour was higher than activated carbon for all of the parameters. As shown in Fig. 1, the percentage reduction increased due to the adsorption capacity of montmorillonite. Similar conclusions were reported by Ahmad [19]. The adsorption of foulants on the surface of montmorillonite is affected by the surface charge of montmorillonite. In addition, the low adsorption capacity of activated carbon was influenced by the particle size. The activated carbon is granular, whereas montmorillonite is a powder. The powder shape has a higher contact area compare to granulated shape. The contact area determines the quantity of the pollutant that can be adsorbed by the adsorbent [7].

3.2. Effect of mixing speed with different contact times (pH 7, 4 g/L and 60 min)

There were two agitation speeds applied in the experiments, that is, 300 and 500 rpm. From Fig. 2, there was no significant difference in the reductions of COD, TSS and colour. By increasing the mixing speed,

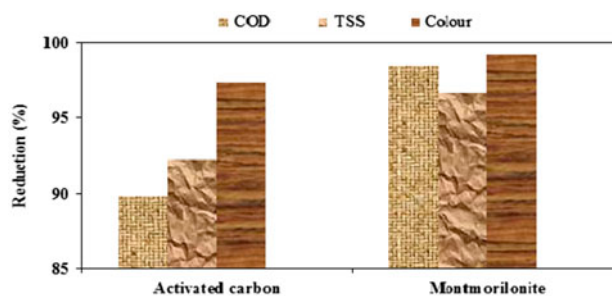


Fig. 1. Comparison of activated carbon and montmorillonite.

the resistance of the surface film is reduced and, therefore, particles were easily attached and adsorbed on the surface of adsorbent. Another reason is that the increasing agitation speed leads to collisions among particles. The collisions crush the particles and break them into smaller particles. The smaller particles enter the pores of the adsorbent more easily. By considering the effect of mixing speed on the particle size, the 300 rpm agitation was chosen to be throughout the study.

3.3. Effect of POME concentration (pH 7, 300 rpm, 4 g/L and 60 min)

Fig. 3 shows the effect of POME concentration on the three parameters: COD, TSS and colour. The POME concentration was diluted from 100 to 25% using ultrapure water. The COD and colour showed a slight decreasing, but the opposite occurred for the TSS. By increasing the concentration of POME, the TSS also decreased. In the solution, suspended solids can easily be seen, which is in contrast to the COD and colour. It is assumed that when the concentration of POME is increased, the suspended solids also increased, while the number of adsorbents remains constant. The adsorbent cannot provide a large number of active sites, so there is a lack of availability of solute adsorption [20]. Consequently, the capacity of the adsorbent to adsorb solids is lowered, so the lowest concentration of POME was the best option.

3.4. Effect of adsorbent dosage (pH 7, 300 rpm and 60 min)

The dosage of the adsorbent is a common factor that influences the success of adsorption. It can be seen from Fig. 4 that increasing the dosage of adsorbent affects the COD and TSS, but not the colour reduction. In fact, the percentage reductions of the parameters were high and reached more than 95%.

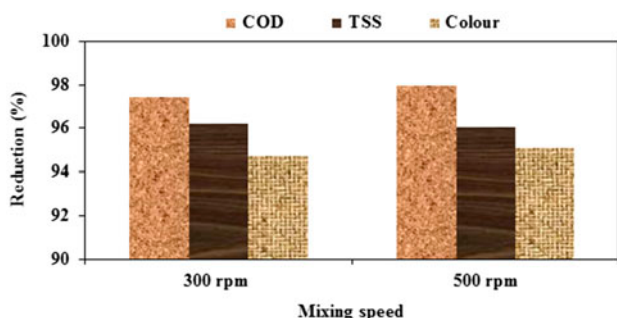


Fig. 2. Effect of mixing speed on COD, TSS and colour reductions.

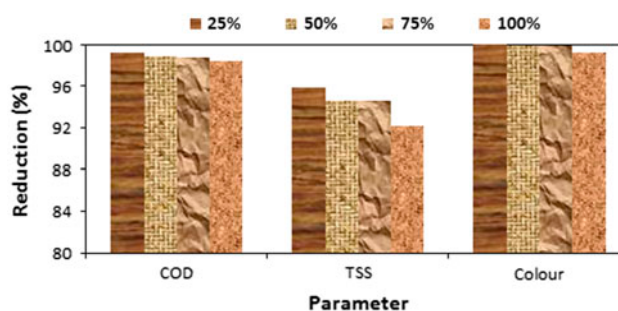


Fig. 3. Effect of POME concentration on COD, TSS and colour reductions.

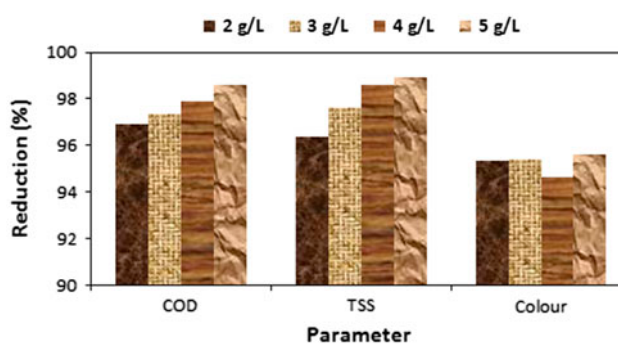


Fig. 4. Effect of adsorbent dosage on COD, TSS and colour reductions.

Adsorption generally correlates to specific surface area phenomena and surface selectivity. The specific surface area is the total surface available for adsorption. The surface selectivity can be classified as hydrophilic or hydrophobic. Montmorillonite is negatively charged, which comes from isomorphous substitution. The negative charge neutralises the cations in the solution, which finally deposits on the surface of the adsorbent. In addition, the additional adsorbent dosage expanded the surface area, which makes a

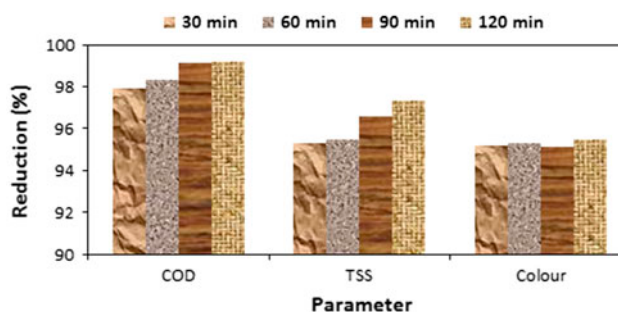


Fig. 5. Effect of contact time on COD, TSS and colour reductions.

larger contact area. Both of the conditions increased the number of dissolved organic molecules to be adsorbed. Based on the results, the optimum adsorbent dosage was determined to be 5 g/L. The results agree with previous studies, showing that increasing the dosage of adsorbent increases the removal of pollutants [8,9,19].

3.5. Effect of contact times (pH 7, 4 g/L and 300 rpm)

Fig. 5 shows the correlation between contact time and the reduction of COD, TSS and colour. As shown, the adsorption of COD, TSS and colour were fast and the equilibria were reached within 90 min. After 90 min, the reduction percentages of COD and colour were slightly increased. In contrast, for the suspended solids, the reduction became higher after 90 min. It is predicted that montmorillonite not only acts as the adsorbent, but also as the coagulant. It makes the particles agglomerate into big particles so that they may be retained by the adsorbent. Based on this result, an optimal contact time of 90 min was the best option.

3.6. Effect of pH (4 g/L, 300 rpm and 90 min)

The effect of pH was investigated by varying the pH value from 3 to 9. As can be seen in Fig. 6, two phenomena were obtained. Firstly, the COD and colour tend to going down after pH 7 and, secondly, the TSS parameter does the opposite. This indicated that the pH has a significant effect on COD, TSS and colour reductions. The effect of pH is concerned with the surface charge and degree of ionisation [21]. Under acidic conditions, there are not sufficient protons on the surface of montmorillonite to adsorb the pollutant from POME. As the pH is increased, most of the charge on the adsorbent surface is deprotonating, thus facilitating the binding attraction between the adsorbent and the particles [22].

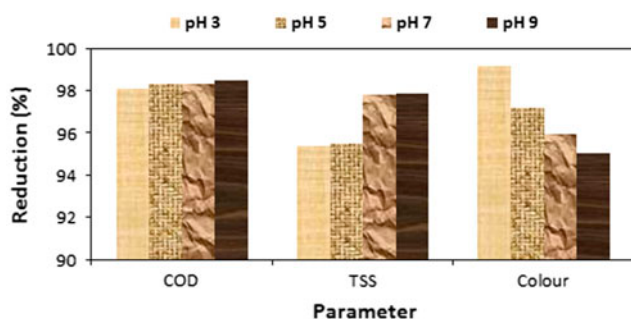


Fig. 6. Effect of pH on COD, TSS and colour reductions.

3.7. The adsorption isotherm model

The adsorption isotherm using montmorillonite can be calculated using Langmuir and Freundlich equations [23], which are usually written as:

$$\frac{C_e}{Q_e} = \frac{1}{Q_m K_1} + \frac{C_e}{Q_m} \quad (3)$$

$$\log Q_e = \log K_f + \frac{1}{n} \log C_e \quad (4)$$

where C_e is the equilibrium concentration of the solution (mg/L), Q_e is the amount of solute adsorbed at equilibrium (mg/g), Q_m is the maximum adsorption capacity (mg/g), $1/n$ is the adsorption intensity, and K_1 and K_f are the Langmuir and Freundlich constants, respectively.

K_1 and Q_m were calculated from the slope and intercept of the plot of $\frac{C_e}{Q_e}$ vs. C_e , whereas K_f and $1/n$ were calculated from the slope and intercept of the plot of $\log Q_e$ vs. $\log C_e$. The constants of the Langmuir and Freundlich models are presented in Table 3. The smaller value of $1/n$ indicates the strong bond between the adsorbate and adsorbent, so it will impact for better adsorption processes [24].

It can be seen in Fig. 7 that the Langmuir model yields a somewhat better fit than the Freundlich model for the removal of COD, TSS and colour. This means that the experimental data can fulfil the Langmuir equation, but not the Freundlich equation. From the figure, we can see that montmorillonite is efficient in adsorbing the pollutant from POME. It can be seen that the maximum adsorption is achieved at equilibrium. Therefore, the adsorption process takes place as a monolayer on the homogeneous, rather than heterogeneous, surface. The monolayer can form if the pore size of the solute is similar to montmorillonite. Solute attaches to the surface of montmorillonite and diffuses into the pores before finally being trapped inside of the adsorbent. The solute will be continuously deposited on the surface of the adsorbent and the layer will become thicker. The interaction force between the solute and the adsorbent means that the solutes cannot leave the adsorbent. This was proven by the essential characteristics of the Langmuir isotherm model, which can be calculated by the following equation:

$$R_L = \frac{1}{1 + K_1 C_0} \quad (5)$$

where C_0 is the initial concentration of solute (mg/L). The R_L values in this experiment were found

Table 3
Langmuir and Freundlich constants for adsorption using montmorillonite

Parameter	Langmuir isotherm model			Freundlich isotherm model		
	K_L (L/g)	Q_m (mg/g)	R^2	K_f (mg/g)	$1/n$ (-)	R^2
COD	1.714286	1428.571	0.9866	1578.338	0.012	0.9899
TSS	0.092652	344.8276	0.899	502.458	0.0617	0.8945
Colour	1.666667	10,000	0.9989	6834.394	0.0027	0.9904

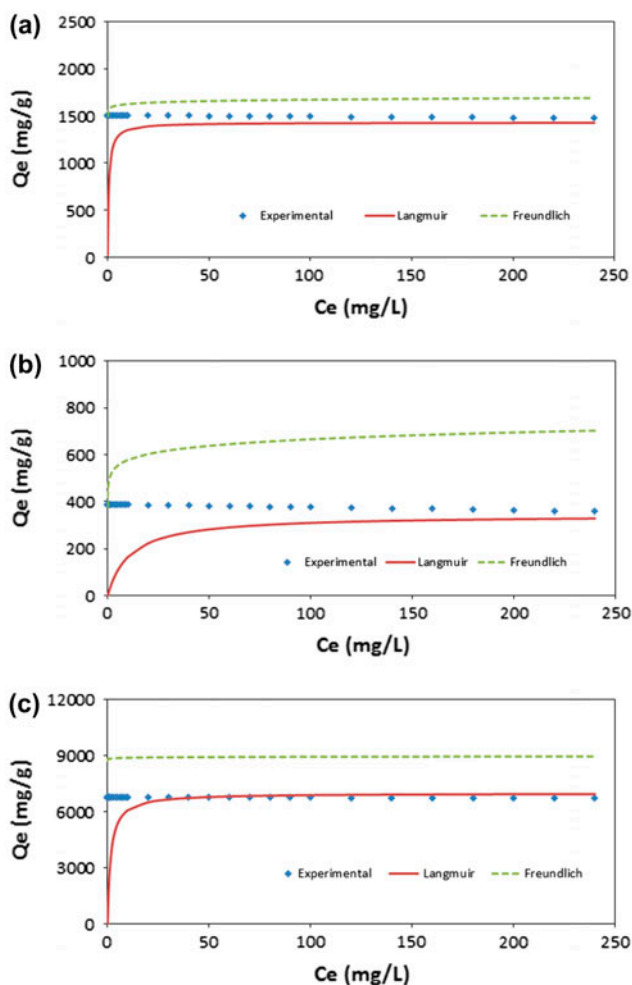


Fig. 7. Experimental data fitting to Langmuir and Freundlich equations for the removal of (a) COD, (b) TSS, and (c) colour.

to be 0.000048, 0.003466 and 0.000011 for COD, TSS and colour, respectively. This indicates that the adsorption process using montmorillonite is irreversible, which means the solute and adsorbent experience strong attractive forces. Therefore, the absorbed solute cannot be removed from the

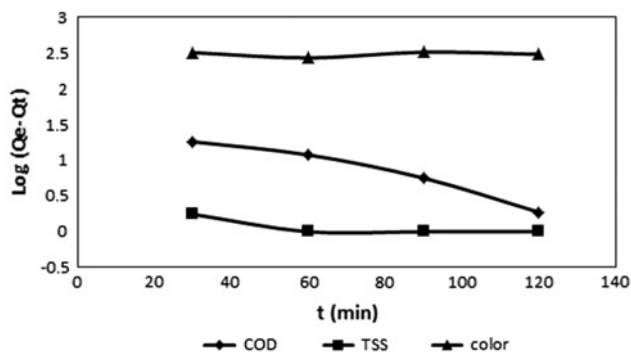


Fig. 8. Pseudo-first-order kinetic model for adsorption using montmorillonite.

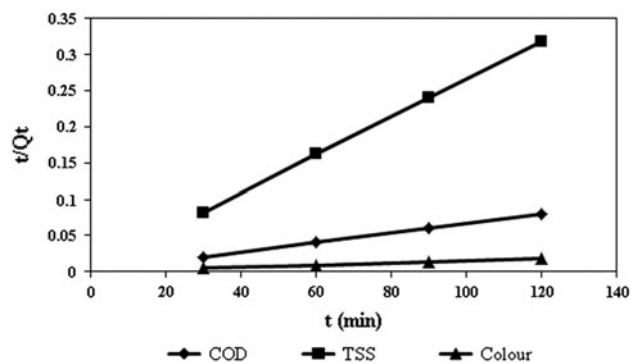


Fig. 9. Pseudo-second-order kinetic model for adsorption using montmorillonite.

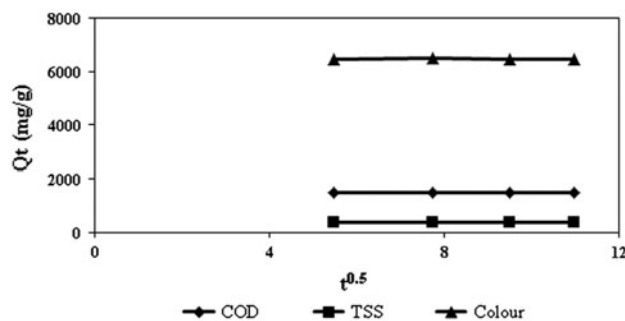


Fig. 10. Intra-particle diffusion model for adsorption using montmorillonite.

Table 4
Comparison of three kinetic adsorption models using montmorillonite

Parameter	$Q_{e, \text{exp}}$	Pseudo-first order			Pseudo-second order			Intraparticle diffusion		
		K_1	$Q_{e, \text{cal}}$	R^2	K_2	$Q_{e, \text{cal}}$	R^2	K_i	C	R^2
COD	1428.57	0.0035	79.01	0.834	1.215	1,428	1	3.615	1452.9	0.891
TSS	333.33	0.0080	2.34	0.881	0.0021	384.61	1	1.467	360.84	0.889
Colour	1.0000	4.6E-5	303.54	0.001	0.004	5,000	1	0.334	6464.1	0.001

adsorbent, except when using a chemically and thermally activated process (regenerative process).

3.8. Kinetic sorption

The mechanism of adsorption can be explained using the pseudo-first-order and pseudo-second-order equations, as follows:

$$\log(Q_e - Q_t) = \log Q_e - \frac{K_1}{2.303} t \quad (6)$$

$$\frac{t}{Q_t} = \frac{1}{K_2 Q_e} + \frac{t}{Q_e} \quad (7)$$

where Q_e (mg/g) and Q_t (mg/g) are the adsorption at equilibrium and time t , respectively. K_1 (1/min) and K_2 (g/mg min) are the rate constants of pseudo-first- and -second-order, respectively. For the pseudo-first-order model, Q_e and K_1 can be determined from the slope and intercept of the plot of $\log(Q_e - Q_t)$ vs. t (Fig. 8), respectively, whereas K_2 can be calculated from the plot of t/Q_t vs. t (Fig. 9).

Furthermore, the adsorption mechanism was also influenced by the diffusion of the solute into the pores of the adsorbent. The intraparticle diffusion model describes the phenomenon, which can be written as the following formula:

$$Q_t = K_i t^{0.5} + C \quad (8)$$

The slope and intercept of the plot of Q_t vs. $t^{0.5}$ (Fig. 10) are used to determine the intraparticle diffusion rate constant, K_i and the boundary layer thickness, C , respectively.

The calculation data are tabulated in Table 4. It can be seen that the pseudo-first-order model is not eligible. Based on the R^2 value, the data were well fitted with the pseudo-second-order model. The $Q_{e, \text{cal}}$ values for the pseudo-second-order model are also close to the $Q_{e, \text{exp}}$. For K_2 , the COD had the highest values

compared with TSS and colour. It was proven by the fact that the percentage of COD reduction was always constant, that is, over 96%, whereas that for TSS and colour tended to fluctuate.

Similar to the pseudo-second-order model, the intraparticle diffusion model also had a similar adsorption capacity (C) with the equilibrium adsorption ($Q_{e, \text{exp}}$). Solutes became attached to the surface of the membrane and diffused to the inside of the adsorbent through the pores. The higher K_i value compared to K_1 and K_2 determines that intraparticle diffusion controls the whole process of adsorption. Therefore, the solute will be trapped inside the pores and cannot be removed by a simple cleaning method. This situation causes the adsorbent to seldom be used repeatedly.

4. Conclusion

The adsorption of POME using montmorillonite has been investigated. Various factors have been applied in the adsorption process. The optimum conditions of mixing speed, POME concentration, adsorbent dosage, contact time and pH value were 300 rpm, 25% POME, 5 g/L, 90 min and pH 7, respectively. From the Langmuir isotherm adsorption equation, the maximum adsorption capacity for the COD, TSS and colour parameters were 1428.6, 333.3 and 10,000 mg/g, respectively. The experimental data were fitted with the pseudo-second-order kinetic and intraparticle diffusion models. The kinetic sorption model followed the sequence of $K_i > K_2 > K_1$, which determined that intraparticle diffusion controls the adsorption process.

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