Microwave-Induced Zinc Chloride Activated Palm Kernel Shell for Dye Removal

(Isirong Sawit Teraktif Zink Klorida dengan Gelombang Mikro Sebagai Penyingkir Warna)

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ABSTRACT

This work is aimed to determine the characteristics of activated carbons derived from palm kernel shell (PKS) by microwave-induced zinc chloride activation for dye removal. Activation was performed in a microwave oven at power intensity of 70% for 10 min. The same procedures were repeated for activation using recycled $ZnCl_2$ solution from the first activation. The activated carbons were characterized according to surface area, morphology, functional groups and batch adsorption. The yield for the first activation was 70.7% with surface area of $858 m^2/g$. It was found that the activated carbon prepared using the recycled $ZnCl_2$ still possesses good surface area for methylene blue removal. The adsorption behaviour of the continuous system was well fitted to and could be satisfactorily described by the Yoon and Nelson model.

Keywords: Adsorption; methylene blue; microwave heating; palm kernel shell; zinc chloride activation

ABSTRAK

Kajian ini bertujuan untuk menentukan ciri karbon teraktif daripada isirong sawit (PKS) dengan pengaktifan zink klorida (ZnCl₂) berbantukan gelombang mikro sebagai penjerap warna. Pengaktifan dilakukan dalam ketuhar gelombang mikro pada keamatan kuasa 70% selama 10 min. Prosedur yang sama diulang bagi pengaktifan menggunakan ZnCl₂ yang dikitar semula daripada pengaktifan pertama. Karbon teraktif dicirikan mengikut luas permukaan, morfologi, kumpulan berfungsi dan penjerapan berkelompok. Hasil untuk pengaktifan pertama ialah 70.7% dengan luas permukaan 858 m²/g. Juga didapati bahawa karbon teraktif yang disediakan dengan ZnCl₂ yang dikitar semula masih mempunyai luas permukaan yang baik untuk penyingkiran metilena biru. Sifat penjerapan sistem berterusan adalah menepati dan boleh digambarkan dengan model Yoon dan Nelson.

Kata kunci: Isirong sawit; metilena biru; pemanasan gelombang mikro; pengaktifan zink klorida; penjerapan

INTRODUCTION

The release of dyes from textile industries into receiving water affects the aquatic creatures because they are highly toxic and carcinogenic (Environmental Protection Agency 1997). Dyes block the passage of sunlight into the stream, thus destroy the life cycle and food chain in the water and disrupt the biodiversity within. As water is a basic requirement in daily life, the deteriorate quality of water brings bad consequences to human being. Activated carbon adsorption is a preferred separation technique over other physico-chemical methods of dye removal because of rich surface and pore properties of activated carbon (Anjaneyulu et al. 2005; Forgacs et al. 2004; Robinson et al. 2001). However, precursors of commercial activated carbon are not renewable, while regeneration of spent activated carbon is relatively expensive. This scenario has triggered searches for new alternative carbonaceous precursors that are abundantly available and low cost. One of the promising candidates under this category is palm kernel shell (Allwar et al. 2008; Guo & Lua 2003; Issabayeva et al. 2006; Tan et al. 2008).

Palm oil mills in Malaysia produce nearly 4.3 million tones of palm kernel shell annually (Husain et al. 2003). Some of the waste is used as fuel to produce process steam or to generate electricity in the mills (Husain et al. 2003; Okoroigwe & Saffron 2012). Yet these only consumed small portion of palm kernel shell, while the remaining large portion is either burned in open air or dumped scattered in areas around the mill. Thus, its conversion into activated carbon could possibly solve the disposal and environmental problems due to unutilized palm kernel shell at the mills (Issabayeva et al. 2006; Tan et al. 2008). Microwave heating is an emerging alternative to substitute conventional heating at high temperature in the preparation of activated carbon (Menendez et al. 2010; Yuen & Hameed 2009). In microwave heating, energy is transferred into heat by the act of microwaves, i.e., heating from the interior of the material through dipole reorientation and conductive loss mechanisms, and so no contact between the material and the heating source is necessary (Metaxas & Meredith 1983; Mullin 1997). Microwave heating in general offers rapid and efficient heat transfer, short treatment time, lower

energy consumption and insignificant release of harmful gas (Menendez et al. 2010; Yuen & Hameed 2009).

The present study aimed to investigate the adsorptive properties of palm kernel shell activated carbon prepared through microwave-induced zinc chloride ($ZnCl_2$) activation for methylene blue dye removal. The effects of using recycled $ZnCl_2$ from the first activation, initial dye concentration and flow rate of column setting were examined and discussed.

MATERIALS AND METHODS

PREPARATION OF ACTIVATED CARBON

Palm kernel shell (PKS) was obtained from Felda Taib Andak Palm Oil Mill, Kulai, Johor. Structural and elemental compositions of PKS are given in Table 1 (Okoroigwe & Saffron 2012).

TABLE 1. Structural and elemental compositions of palm kernel shell

Structural composition (%)	
Lignin	52
Cellulose	7
Hemicellulose	26
Moisture	6
Ash	9
Elemental (%)	
Carbon	50
Hydrogen	5.6
Nitrogen	0.72
Oxygen[a]	35

[a] by difference

Zinc chloride ($ZnCl_2$) was dissolved and mixed with 10 g palm kernel shell (PKS) at weight ratio of 1. One-step chemical activation was carried out in a microwave oven at power intensity of 70% for about 10 min until the mixture was completely dried. The resultant activated carbon was washed using distilled water in a soxhlet unit to recover zinc chloride for another activation of 10g raw PKS. Same activation procedures were repeated. The activated carbons were designated as Z-PKS and RZ-PKS for activation using fresh and the recycled $ZnCl_2$, respectively. The activated carbons were dried in oven prior to be used for characterization and adsorption.

CHARACTERIZATION OF ACTIVATED CARBON

Surface area of activated carbons was measured using surface area analyzer (Micromeritics Pulse ChemiSorb 2705, USA) at liquid nitrogen temperature of 77K, while surface morphology was determined using SEM instrument (Philips XL 40, Netherlands). The surface functional groups were determined using FTIR instrument (Perkin Elmer Spectrum 2000 Explorer, USA) to obtain peaks at different wavelengths.

Methylene blue ($C_{16}H_{18}ClN_3S.3H_2O$, MW=373.9 g/mol), one of the widely used dyes in textile industries was employed as pollutant probe in adsorption. In batch adsorption, 0.1 g of activated carbon was added into conical flasks containing 50 mL of methylene blue solution at concentrations of 10, 20 and 50 ppm. The solution pH was not adjusted, and measured as 5.8 ± 0.1 for all initial concentrations studied. The flasks were sealed, and the mixtures were allowed to equilibrate on orbital shaker at $28\pm1°C$ and 90 rpm for 72 h. There after, the solutions were filtered and the residual concentrations were determined using Visible Spectrophotometer (Biochrome Libra S6, UK) at a wavelength of 690 nm.

COLUMN ADSORPTION

A column having a length and diameter of 20 and 2.5 cm was utilized for bench-scale continuous adsorption. The depth of the adsorbent was fixed at 2.5 cm (4.1g). The column was sealed to prevent methylene blue solution from flowing out through the top and bottom openings. Peristaltic pump was used to pump the liquid into the column from the bottom and leaving the column top. Initial concentration of methylene blue was fixed at 10 ppm and the continuous adsorption was performed at room temperature $28 \pm 1°C$, and two different flow rates, i.e., 73 and 166 mL/min. The effluent was collected every 2 min to determine the residual concentration.

RESULTS AND DISCUSSION

CHARACTERISTICS OF ACTIVATED CARBON

Table 2 shows the yield and surface area of palm kernel shell activated carbons.

TABLE 2. Yield and surface area of activated carbons

	Z-PKS	RZ-PKS
Yield (%)	70.7	51.4
Surface area (m²/g)	858	345

The yield of Z-PKS is greater than that of RZ-PKS. This could be explained by the fact that the release of volatile matter from the precursor is inhibited when the ratio of activating agent to precursor is higher (Deng et al. 2009). It is believed that the ratio of recycled $ZnCl_2$ to activate the RZ-PKS is lesser than 1 possibly due to the amount of $ZnCl_2$ that was used up and evaporated from the first activation. It is also likely that certain amount of Zn is trapped and retained on the surface of Z-PKS, which lessen the original weight of $ZnCl_2$ available for reuse, thus resulting in a lower yield of RZ-PKS (Allwar et al. 2008).

From Table 2, the values of surface area were recorded as 858 and 345m²/g for Z-PKS and RZ-PKS, respectively.

Clearly the surface area of Z-PKS is greater than that of RZ-PKS. This proved that $ZnCl_2$ impregnation ratio plays an important role during the activation of PKS, wherein its amount determines the surface area of activated carbon. On the other hand, a lower surface area of RZ-PKS is likely due to lower impregnation ratio of $ZnCl_2$ which was not fully recovered from the first activation. Wang et al. (2009) and Zaini et al. (2009) reported that the increase of $ZnCl_2$ ratio in activation has resulted in the increase of surface area through the development of mesoporosity.

The surface area of activated carbon reported in this work is comparable to that of PKS activated carbons prepared using conventional heating (Allwar et al. 2008; Guo & Lua 2003; Issabayeva et al. 2006; Tan et al. 2008). Yet, microwave heating offered a lot shorter time for activation and the yield is also higher compared to conventional heating (Menendez et al. 2010; Yuen & Hameed 2009; Zaini et al. 2009). Similar works on microwave-assisted activated carbon preparation using different carbonaceous precursors are given in Table 3.

In general, the mixture of precursor and activating agent possesses good polar and receptor properties that are readily viable for microwave heating and activated carbon preparation. Unlike earlier studies in Table 3, attempt was made in this work to utilize existing domestic microwave with no modification and the activation occurred in the absence of inert carrier up until the mixture was completely dried. Although the period taken for activation is comparable, it is proposed that inert carrier (such as N_2) may not necessarily be required in microwave-assisted activation for reducing burn-off as in conventional heating. Using a current approach, we reported a comparable surface area of activated carbon with a better yield (Table 2).

Figure 1 shows the SEM images of activated carbons, where both activated carbons exhibit a well-developed porosity with considerable textures of micropores and mesopores on their external surface. These are in agreement with their respective values of surface area. The cavities serve as a passageway to the abundant micropores and mesopores in the internal structure of activated carbon. Under the same magnification, the SEM image for Z-PKS shows rich pores and cavities with some external pores greater than 2 nm due to higher impregnation ratio in activation (Zaini et al. 2009).

Table 4 summarizes the peaks of FTIR spectra for Z-PKS and RZ-PKS. The peaks qualitatively represent available functional groups on the surface of activated carbon.

Obviously, both activated carbons possess the same spectra which indicate identical surface properties within their structure. The C–H stretching vibration at wavelength of around 2950 cm^{-1} was not found because the hydrogen element is probably removed to a large extent during activation process. It also signifies that the aliphatic chains have satisfactorily transformed into the graphitic structures of activated carbon. Functional groups such as hydroxyl, lactonic and carbonyl are present on both activated carbons due to oxidation of carbon surface in activation. The

TABLE 3. Precursors used in microwave-assisted activated carbon preparation

Precursor	Activating agent	Activation time (min)	Surface area (m^2/g)	Reference
Pine wood powders	$ZnCl_2$	10	1459	Wang et al. (2009)
Sewage sludge	H_3PO_4 $ZnCl_2$	10	291 377	Wang et al. (2011)
Oil palm fiber	KOH	5	708	Foo & Hameed (2011)
Cotton stalk	$ZnCl_2$	9	794	Deng et al. (2009)
Pineapple peel	KOH	6	1006	Foo & Hameed (2012)
Oil sands coke	KOH	10	1131	Chen & Hashisho (2012)
Palm kernel shell	$ZnCl_2$	10	858	Present study
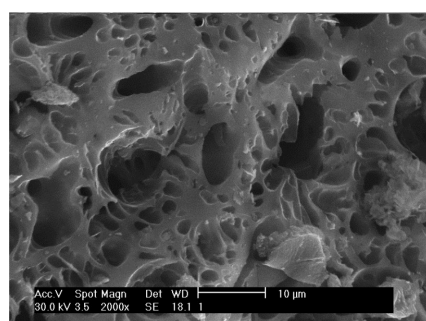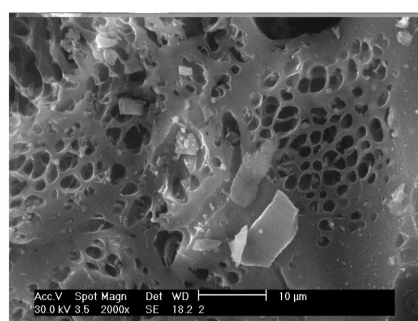
(a)   (b)

FIGURE 1. SEM images for (a) Z-PKS and (b) RZ-PKS

presence of aliphatic chloro compound, C–Cl stretching at wavelength of 750 cm^{-1} could be attributed to the reaction between the activating agent and the carbon surface.

EFFECT OF ACTIVATION USING RECOVERED ZnCl$_2$

Attempt to activate PKS using recovered ZnCl$_2$ was demonstrated in this work. As already highlighted, such activation was feasible even though the yield and surface area are lower compared to the first activation.

Figure 2 shows the degradation profile of ZnCl$_2$ against temperature that was generated using a Seiko EXSTAR6000 TG/DTA6200 instrument.

Clearly, rapid weight loss due to evaporation started at 500°C and almost all ZnCl$_2$ is completely degraded at 550°C. Conventional heating for activated carbon preparation was commonly established at temperatures between 500 and 900°C (Allwar et al. 2008; Zaini et al. 2009) thus the recovery of ZnCl$_2$ is almost impossible to happen. While, the temperature at which the mixture was completely dried via microwave-assisted process is expected to be relatively lower than that in conventional

heating, thus it is likely to recover part of the used ZnCl$_2$ for subsequent activation. The recovered solution was still sufficient to be reused in activation process, where considerable amount of pores and cavities can be found on the surface of RZ-PKS (Figure 1(b)).

BATCH ADSORPTION

Figure 3 shows the linear isotherm of methylene blue removal onto activated carbons. At equilibrium, no significant change was observed on the solution pH and the values were recorded as 5.9±0.1. Both activated carbons show an increase profile with increasing equilibrium concentration, which indicates that more sites are still vacant to adsorb methylene blue dye and the saturation has not already been achieved. At higher concentration, activated carbons are expected to adsorb greater amount of methylene blue dye up until a plateau is attained. From Figure 3, it was found that Z-PKS still agreed with the Henry's Law even at higher concentration of 50 ppm. Moreover the intensity of methylene blue adsorption towards Z-PKS is 6.7 times higher than that of RZ-PKS, which
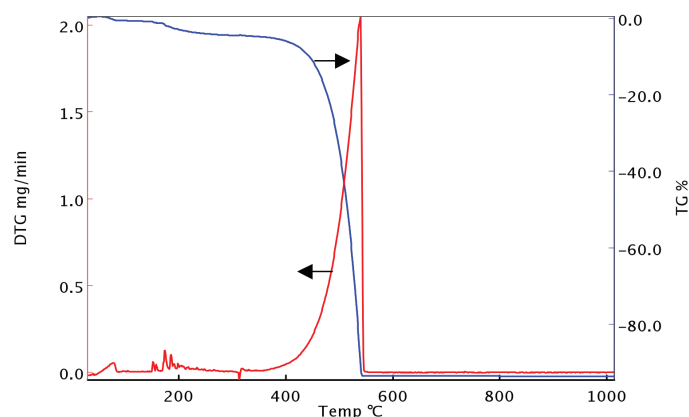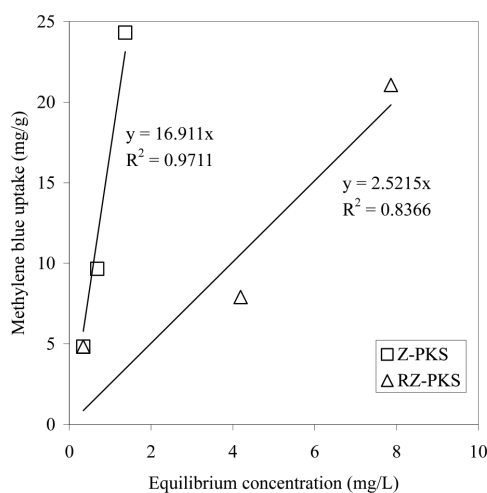

FIGURE 2. TG/DTA profile of ZnCl$_2$

FIGURE 3. Linear equilibrium isotherm of methylene blue removal on to activated carbons

corresponds to the rich pore textures and surface area of the former (Table 1 and Figure 1).

Figure 4 presents the uptake capacity of methylene blue onto activated carbons at different initial concentrations. At lower methylene blue concentration of 10 ppm, Z-PKS and RZ-PKS exhibited same adsorption capacity of 5 mg/g. This is equivalent to 97% removal of methylene blue. The uptake capacity of both activated carbons increased with increasing initial concentration, which signifies the role of pores to accommodate the adsorption of methylene blue. However, the uptake by RZ-PKS was somewhat lower as the initial concentration increases to 50 ppm, due to smaller number of available sites for adsorption. Undoubtedly, the adsorption performance of RZ-PKS was not as good as Z-PKS because the surface area of the former is about half of the latter. Yet, the percent removal of RZ-PKS was still good at about 80% for higher methylene blue concentrations of 20 and 50 ppm.

CONTINUOUS ADSORPTION

Breakthrough curve The uptake behaviour of methylene blue solution in a fixed bed is commonly expressed as a plot of normalized concentration (ratio of effluent concentration over initial concentration) against time to obtain a breakthrough curve. The maximum column capacity, q_{Total} can be expressed as,

$$q_{Total} = \frac{QA}{1000},$$ (1)

where Q (mL/min) is the flow rate and A is the area under the breakthrough curve. The equilibrium uptake, q_e (mg/g) is calculated from equation below,
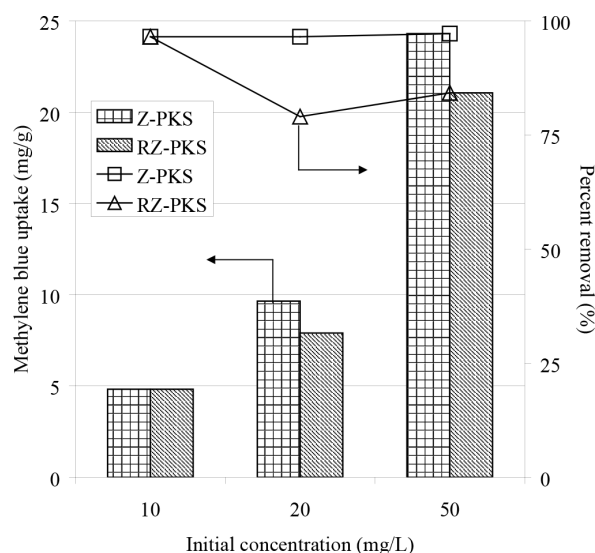
$$q_e = \frac{q_{Total}}{m},$$ (2)

FIGURE 4. Uptake capacity and percent removal of methylene blue at different initial concentrations

where m (g) is the weight of adsorbent in column. The amount of methylene blue supplied to the column, W_{Total} (mg) is calculated as,

$$W_{Total} = \frac{C_0Qt}{1000},$$ (3)

where C_0 (mg/L) is the influent concentration and t (min) is the total time for column operation. The percent removal of methylene blue is determined as the ratio of q_{Total} over W_{Total}. The parameters of column adsorption are summarized in Table 5.

Figure 5 shows the breakthrough of continuous adsorption of methylene blue onto Z-PKS bed.

From Figure 5, the breakthrough curves demonstrate a sharp front of the curve followed by broadening of

TABLE 4. Assignments of functional groups in FTIR spectra

Z-PKS	RZ-PKS	Functional group
3400 cm^{-1}	3380 cm^{-1}	Hydroxy group, O–H stretching
2350 cm^{-1}	2350 cm^{-1}	Triple bonded carbon, C≡C stretching
1600 cm^{-1}	1590 cm^{-1}	Lactonic and carbonyl group, C=O stretching
1220 cm^{-1}	1150 cm^{-1}	Tertiary alcohol, C–O stretching
750 cm^{-1}	750 cm^{-1}	Aliphatic chloro compound, C–Cl stretching

TABLE 5. Parameters of column adsorption at different flow rates

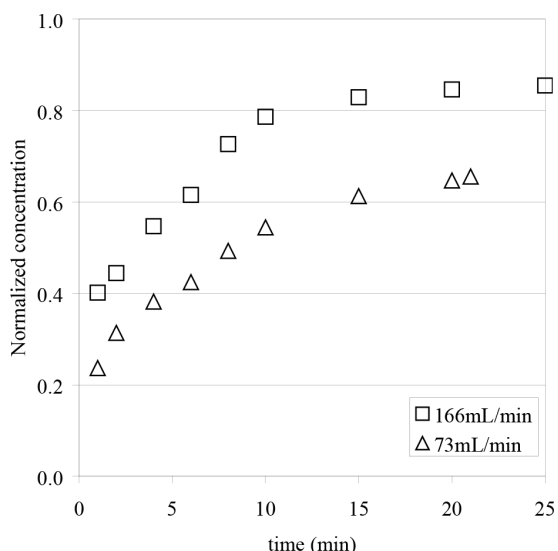| Parameters	Flow rate 73 mL/min	Flow rate 166 mL/min
A	10.6	18
t (min)	21	25
q_{Total} (mg)	0.772	2.99
q_{eq} (mg/g)	0.188	0.730
W_{Total} (mg)	15.3	41.5
Percent removal (%)	5.04%	7.20%

FIGURE 5. Effect of different flow rates on the breakthrough of methylene blue adsorption onto Z-PKS bed

tail. It could be explained by mass transfer resistance of adsorbent-phase that dominates the overall adsorption rate (Helfferich & Carr 1993). The column was found to perform better at flow rate of 73 mL/min because longer breakthrough and exhaustion time could be achieved. On the other hand, higher flow rate caused faster exhaustion of Z-PKS due to insufficient residence time for solutes to diffuse into the pores of Z-PKS. Hence, the solutes left the column before the equilibrium is attained. Similar trend of breakthrough was reported elsewhere (Aksu & Gonen 2004; Kumar et al. 2005; Tan et al. 2008; Yahaya et al. 2011).

Kinetics models Kinetics models are used to describe the dynamic behaviour of the column adsorption and to predict the parameters of breakthrough. In this work, two models namely Adam's-Bohart and Yoon and Nelson were used to describe the column adsorption data.

Adam's–Bohart model (Bohart & Adams 1920) described the initial part of the breakthrough curve and is given as,

$$\ln\left(\frac{C_t}{C_0}\right) = k_{AB}C_0 t - \frac{k_{AB}N_0 Z}{L}, \tag{4}$$

where k_{AB} (mL/mg. min) is the kinetic constant, L (cm/min) is the linear velocity, Z (cm) is the bed depth of the column and N_0 (mg/mL) is the saturation concentration. Yoon and Nelson model (Yoon & Nelson 1984) is used to describe the decrease in adsorption rate that is proportional to the interaction probability between adsorbate adsorption and adsorbate breakthrough on the adsorbent. The model is expresses as below,

$$\ln\left(\frac{C_t}{C_0 - C_t}\right) = k_{YN}t - k_{YN}\tau, \tag{5}$$

where, k_{YN} (min^{-1}) is the rate velocity constant and τ (min) is the time required for 50% adsorbate breakthrough. All unknown parameters from the models were solved using *Solver*-add in of Microsoft Excel for lowest sum of squared error (SSE) and optimum coefficient of determination (R^2).

Table 6 summarizes the constants of breakthrough models at different flow rates.

The adsorption data were not adequately fitted to Adam's-Bohart model because of fairly poor values of regression coefficient. However, the adsorption of methylene blue onto Z-PKS could be satisfactorily described by Yoon and Nelson model. From Table 6, the rate velocity constant, k_{YN} increased with increasing flow rate, but the time required for 50% adsorbate breakthrough, τ decreased. The τ values from the model are in agreement with the corresponding values from Figure 5.

Because there was more contact time for mass transfer to happen when the flow rate is lower, more methylene blue solutes were able to be adsorbed by Z-PKS bed, thus the value of τ is higher. Increased in solution flow rate reduced the residence time of solutes in the column, thus caused the adsorbate to reach 50% adsorbate breakthrough earlier. Similar column behaviour has been reported in much of published literature (Nwabanne & Igbokwe 2012;

TABLE 6. Parameters for breakthrough models at different solution flow rates

Parameter (unit)	Flow rate (mL/min)	
	73	166
Adam's-Bohart model		
k_{AB} (mL/min.mg)	4.27	2.97
N_0 (mg/mL)	1.67	3.33
SSE	0.172	0.175
R^2	0.794	0.735
Yoon and Nelson model		
k_{YN} (min^{-1})	0.0809	0.0928
τ (min)	10.87	1.28
SSE	0.325	0.875
R^2	0.900	0.844

Singh et al. 2009; Tan et al. 2008). Agreement with Yoon and Nelson model proved that the adsorbent-phase mass transfer resistance dominated the overall adsorption rate.

CONCLUSION

Palm kernel shell based activated carbon was prepared through microwave-induced zinc chloride activation. The use of recovered zinc chloride for activation was feasible, and the adsorptive properties of activated carbon are fairly good for methylene blue removal. Surface area of activated carbon produced using recovered zinc chloride is lower probably due to lower impregnation ratio. Column adsorption of methylene blue onto activated carbon suggested that the mass transfer resistance of adsorbent-phase dominated the overall adsorption rate and this behaviour could be adequately described by Yoon and Nelson model.

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