

CARBONISATION-ACTIVATION OF OIL PALM KERNEL SHELL TO PRODUCE ACTIVATED CARBON AND METHYLENE BLUE ADSORPTION KINETICS

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ABSTRACT

The carbonisation-activation system was developed to produce activated carbon from oil palm kernel shell (OPKS). The OPKS was carbonised at 500°C for 3 hr in an electric vertical reactor followed by steaming at 700°C for another 3 hr in the same reactor. The process showed significant results with a high activated carbon yield of 32%, high fixed carbon content of 88.6% with Brunauer-Emmett-Teller (BET) surface area of 305.67 m² g⁻¹. The OPKS-activated carbon was further tested to remove methylene blue. It could adsorb up to 99.7% of methylene blue using only 0.6 g litre⁻¹ dosage of OPKS-activated carbon, for 24 hr of treatment time. The results have been correlated in the Freundlich isotherm which was well fitted to the experimental data over the methylene blue experimental concentration range with correlation coefficients of R²=0.992.

Keywords: oil palm kernel shell, carbonisation-activation, bioadsorbent, activated carbon.

Date received: 12 October 2017; **Sent for revision:** 22 October 2017; **Received in final form:** 18 January 2018; **Accepted:** 5 July 2018.

INTRODUCTION

Oil palm kernel shell (OPKS) is one of the major sources of oil palm biomass with >100 000 t being produced by the palm oil mills every year in Malaysia and expected to increase by 2020 (National Innovation Agency of Malaysia, 2013). In general, the OPKS has a high density of 0.6 g cm⁻³ with hard and tough structure due to high composition of lignin and carbon. These criteria are very important characteristics for the production of

activated carbon (AC) (Hidayu and Muda, 2016). The production of AC from OPKS has been the subject of interest in recent years due to a low cost and abundance generation of OPKS in the palm oil mills. The OPKS-AC is considered to be one of the most commonly used AC as bioadsorbent in various applications, especially in water and wastewater treatment as it has large surface area and able to remove the target compounds from aqueous solution effectively (Nor Faizah *et al.*, 2016; Hidayu and Muda, 2016; Ismaiel *et al.*, 2013).

In the current practice, AC including OPKS-AC is produced using the two-stage processes, *i.e.* carbonisation and activation. Firstly, the feedstock material is carbonised in the absence of oxygen at temperature of <800°C followed by the activation of the carbonised material at >900°C using chemical and/or physical agent. These two processes are usually carried out in two separate reactors, which contribute to a high energy usage, long processing duration and high cost for materials and apparatus

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(Choi *et al.*, 2015; Hidayu and Muda, 2016). Chemical activating agent can be carried out in a single step by combining the carbonisation and activation processes; however chemical process significantly contributes to the environmental pollution (Marrakchi *et al.*, 2017; Singh *et al.*, 2017). Physical activating agent using steam or CO₂ could be considered as alternative since it is cleaner and easier to handle than chemical process. Physical activation process could also produce AC with high surface area when heated at high heating temperatures of >800°C in a single step process ranging from 800-1200 m² g⁻¹ (Herawan *et al.*, 2013; Sierra *et al.*, 2017; Shoaib and Al-Swaidan, 2015; Sun and Jiang, 2010; Nur Sulihatimarsyila *et al.*, 2017).

Therefore, this study aims to develop a carbonisation-activation system with physical activation using steam in order to produce AC from OPKS. The carbonisation-activation system is operated by conducting steam activation process after carbonisation stage without terminating the operation. The OPKS-AC was fully characterised and subsequently tested as a bioadsorbent for methylene blue removal. Isotherms for the adsorption of methylene blue by OPKS-AC were measured and fitted to three different isotherm equations to determine the best isotherm model to represent the experimental adsorption data.

MATERIALS AND METHODS

Raw Materials

Raw OPKS with particle size of 6-15 mm was obtained from the Ulu Kanchong Palm Oil Mill, Ulu Kanchong Estate, Negeri Sembilan, Malaysia. The OPKS obtained was sun dried until the moisture content reached <10% prior to carbonisation-activation process (Nahrul Hayawin *et al.*, 2017).

Carbonisation-activation Process

About 500 g of OPKS was fed into an electric vertical reactor with a length of 127 cm and an internal diameter of 30 cm (Figure 1). The carbonisation process was started by heating externally at the temperature of 500°C for 3 hr, controlled by a K-type thermocouple placed inside the reactor. The activation process was carried out after the 3 hr of carbonisation process by applying steam at 700°C, produced from a steam generator and piped into the reactor for 3 hr. The steam pressure was set at 2.50 psi. The activation temperatures were automatically recorded at an interval time of 60 s using a data logger. The average temperature was taken for at least three runs of carbonisation-activation process. The gaseous emitted during the process was directed via a pipe at the bottom of the reactor before

being discharged through a stainless steel chimney. At the end of the experiment, the OPKS-AC was removed from the reactor, oven dried at 105°C and ground into powder size using a heavy duty grinder (PHILIPS, US) for subsequent analyses and uses.

Characterisation of AC

The moisture content, volatile matter, fixed carbon, and ash content of OPKS-AC were determined using a thermogravimetric analyser (TGA) (Mettler Toledo, TGA/SDTA 851, USA). The compositions of carbon, hydrogen, nitrogen and sulphur in the OPKS-AC were determined using a CHNS analyser (LECO, CHNS932, USA). The higher heating value (HHV) of OPKS-AC was determined by burning 0.5 g of sample in an adiabatic oxygen bomb calorimeter (Parr, 1341 Plain Jacket Calorimeter, USA). The Brunauer-Emmett-Teller (BET) surface area and pore structure characteristic of OPKS-AC were determined using nitrogen adsorption at -196°C (ASAP2020 Micromeritics, New York, USA). The BET surface area (S_{BET}) was calculated from the isotherms using density functional theory (Lastoskie *et al.*, 1993), while total pore volumes (V_{TOTAL}) were estimated from the liquid pore volumes of N₂ at a high relative pressure ($P/P_0 = 0.99$). The micropore volume (V_{MICRO}) and micropore specific surface area (S_{MICRO}) were determined based on the t-plot method (Rouquero *et al.*, 1999; Barrett and Halenda, 1951).

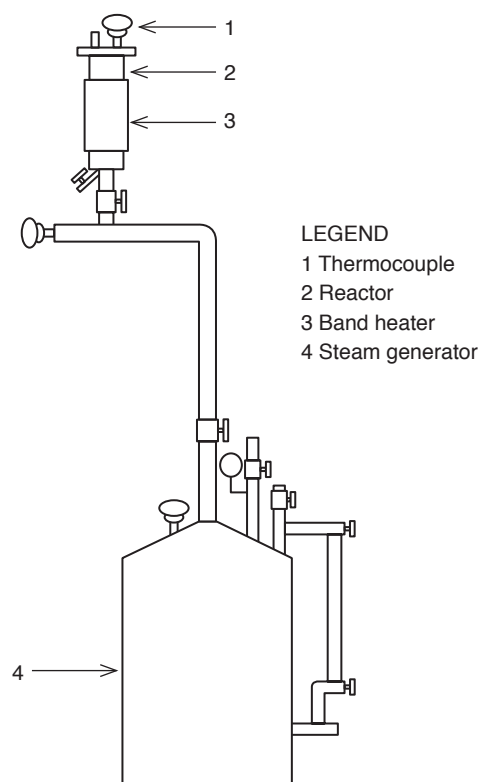


Figure 1. The electric vertical reactor used for the production of activated carbon from oil palm kernel shell (OPKS).

Adsorption of Methylene Blue

Methylene blue was purchased from Sigma-Aldrich with colour content $\geq 82\%$ and initial pH of 7. The adsorption of methylene blue by OPKS-AC was conducted using 0.1 g of OPKS-AC into 25 ml of 500-2000 mg litre⁻¹ of methylene blue prepared in 100 ml of Erlenmeyer flasks. The process was conducted for 24 hr at room temperature of 25°C. Then, the samples were separated by filtration using a vacuum pump. The methylene blue concentrations were determined using a UV-Vis spectrophotometer at a wavelength of 665 nm with the $R^2 = 0.999$. The amount of methylene blue adsorbed onto activated carbon (q_e) and the percentage of methylene blue removal ($R(\%)$) were calculated using Equations (1) and (2).

$$q_e = \frac{V(C_0 - C_e)}{W} \quad \text{Equation (1)}$$

$$R(\%) = \frac{V(C_0 - C_e)}{C_0} \times 100 \quad \text{Equation (2)}$$

where C_0 and C_e are the initial and equilibrium concentrations of methylene blue in mg litre⁻¹, respectively, V is the volume of the solution (litre), and W is the mass of adsorbent (g).

In order to evaluate the effect of adsorbent dosage, four batch experiments were carried out by adding different amounts of OPKS-AC (0.20-0.80 g) into 1000 ml of 500 mg ml methylene blue solution.

Sorption Isotherm Models

The sorption equilibrium data of methylene blue onto OPKS-AC was analysed following the Freundlich, Langmuir and Temkin isotherm models. The linear form of Freundlich's isotherm model is given by Equation (3):

$$\text{Log } q_e = \text{Log } q_e + \frac{1}{n} \text{Log } C_e \quad \text{Equation (3)}$$

where k_f [(mg g⁻¹) (litre g⁻¹)^{1/n}] and n are the Freundlich constants that are related to the adsorption capacity and intensity, respectively. The Freundlich constants k_f and n can be calculated from the slope and intercept of the linear plot, with $\log q_e$ versus $\log C_e$.

The linear form of Langmuir's isotherm model is given by Equation (4).

$$\frac{C_e}{q_e} = \frac{1}{bq_m} + \frac{C_e}{q_m} \quad \text{Equation (4)}$$

where b (litre mg⁻¹) is the Langmuir constant related to the rate of adsorption. The essential characteristics of the Langmuir isotherm can also

be expressed in terms of a dimensionless constant separation factor or equilibrium parameter, R_L , which is defined in Equation (5).

$$R_L = \frac{1}{bC_e} \quad \text{Equation (5)}$$

where b is the Langmuir constant and C_0 is the initial concentration of MB. The value of R_L indicates the type of the isotherm to be either unfavourable ($R_L > 1$), linear ($R_L = 1$), favourable ($0 < R_L < 1$) or irreversible ($R_L = 0$).

The linear form of Temkin isotherm can be expressed in Equation (6).

$$q_e = B \log kt + B \log C_e \quad \text{Equation (6)}$$

where $B = \frac{RT}{b}$ represents heat of adsorption, T is the absolute temperature in Kelvin and R is the universal gas constant, $\frac{1}{b}$ indicates the adsorption potential of the adsorbent while k_t (litre mg⁻¹) is the equilibrium binding constant corresponding to the maximum binding energy. The plot of q_e versus $\log C_e$ enables the determination of isotherm constants k_t and B .

RESULTS AND DISCUSSION

Temperature Profile during Carbonisation-activation Process

Figure 2 shows the temperature profile during carbonisation-activation process of OPKS, and it indicates that carbonisation process took place at minutes 90-270 and while activation process at minutes 300-540. The temperature was taken in average of at least three runs of carbonisation-activation process. The temperature in the electric vertical reactor gradually increased from 0°C to 500°C (heating rate 200°C/30 min) when the heat was introduced within the first 90 min (Figure 2). During carbonisation process at temperature between 300°C-500°C, OPKS released more volatiles compounds as cellulose and lignin content is decomposed at temperature of $>400^\circ\text{C}$ (Choi *et al.*, 2015), therefore, the carbon content was increased. For activation process, the temperature was raised from 500°C to 700°C within 90 min and remained constant until minute 540, before the process was stopped by turning off the steam generation. During this process, more pores were developed and the existing pores were enlarged. The tar that has been filled into the pores during carbonisation process was also removed during the activation process, which had improved the total number of pores and subsequently increased the surface area of AC produced (Abdullah *et al.*, 2011). The feasibility study gave significant results where overall time taken to produce OPKS-AC was only 6 hr with the

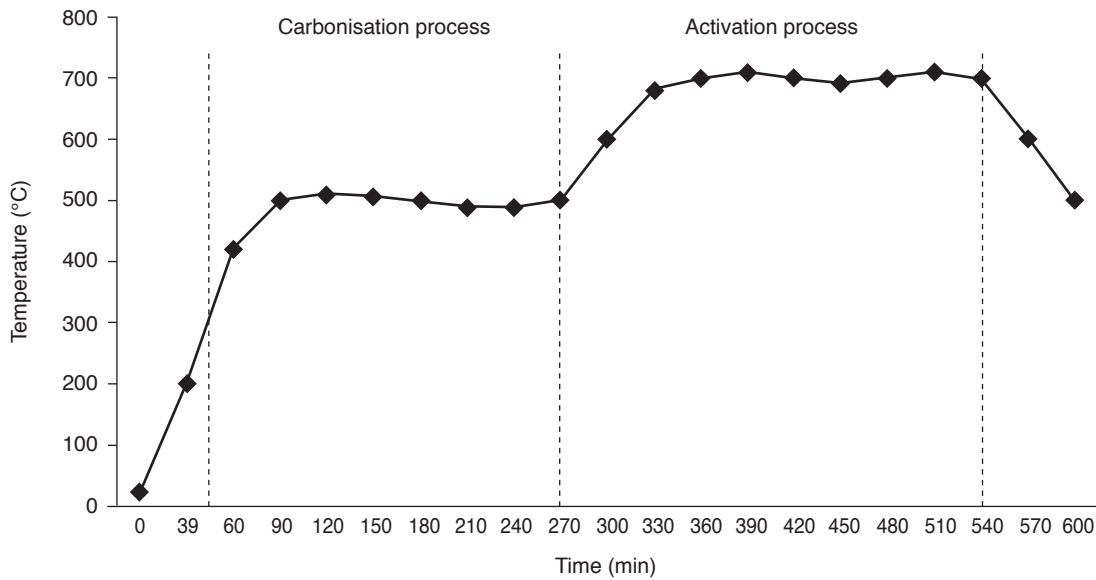


Figure 2. The temperature profile during carbonisation-activation of oil palm kernel shell (OPKS) using electric vertical reactor.

lowest maximum activation temperature at 700°C as compared to the other studies (Sawant *et al.*, 2017; Selvaraju and Bakar, 2017).

Characteristics of OPKS-AC

Proximate and ultimate analysis. An earlier analysis shows that the volatile matter, ash and fixed carbon contents of raw OPKS are 53.40%, 0.87% and 18.84%, respectively (Nahrul Hayawin *et al.*, 2017). After the carbonisation-activation process of OPKS, the OPKS-AC had the highest fixed carbon content of 88.6% and the lowest volatile matter of 3.5% as compared to other studies (Table 1). This phenomenon is due to the rapid release of volatile matter content at high temperature during steam activation process (Arami-Niya *et al.*, 2012). The OPKS-AC also has the lowest ash content of 2.6%, which is ideal for producing AC with highly porous

structure (Abdullah *et al.*, 2011). The results of the proximate and ultimate analyses show that the OPKS-AC produced through the carbonisation-activation system is comparable to AC reported in other studies as shown in Table 1.

Pore characteristics of OPKS-AC. Table 2 shows that the OPKS-AC produced through carbonisation-activation system has the BET surface area, micropore surface area, micropore volume and total volume lower than commercial AC purchased from Sigma. However, the OPKS-AC produced in this study still has the characteristics in the range of standard limit for commercial AC set by gold processing industry, Norit-CAC-Au (Hidayu and Muda, 2016; Ismaiel *et al.*, 2013; Jia and Lua, 2008). According to Buah and Kuma (2016), pore volume, surface area and pore size distribution are important quantitative characterisation of the AC

TABLE 1. PROXIMATE AND ULTIMATE ANALYSES OF OIL PALM KERNEL SHELL-ACTIVATED CARBON (OPKS-AC)

Analysis	OPKS-AC (This study)	Other AC	References
Proximate (%)			
Moisture content	4.3±0.3	1.9-4.6	Arami-Niya <i>et al.</i> (2010)
Ash content	2.6±0.2	2.6-10.6	Choi <i>et al.</i> (2015); Arami-Niya <i>et al.</i> (2010)
Volatile matter	3.5±0.9	10.2-45.9	Arami-Niya <i>et al.</i> (2010)
Fixed carbon	88.6±2.4	44.5-83.0	Arami-Niya <i>et al.</i> (2010)
Ultimate (%)			
C	78.5±4.6	59.6-85.0	Hidayu and Muda (2016); Rugayah <i>et al.</i> (2014); Arami-Niya <i>et al.</i> (2010)
H	2.17±0.0	0.5-4.1	Hidayu and Muda (2016); Rugayah <i>et al.</i> (2014); Arami-Niya <i>et al.</i> (2010)
N	0.41±0.0	0.02-2.9	Hidayu and Muda (2016); Arami-Niya <i>et al.</i> (2010)

porosity. It should also be noted that this OPKS-AC was produced within a short processing duration of 3 hr, at a relatively low activation temperature of 700°C and using carbonisation-activation system without involvement of chemical agent. Besides, this process also produced high yield of OPKS-AC (32%) which is higher than those reported in the literature as shown in *Table 3*. Higher AC yields correspond to steam activation of biomass materials, while chemical activation usually produces lower AC yield. Increasing the activation temperature will increase the surface area by enlarging the existing pores as well as creating new pores. However, too high an activation temperature will decrease the AC yield because it will enhance the evaporation of volatile compounds (Shoaib and Al-Swaidan, 2015). In addition, the resulted surface area for

OPKS-AC is shown to be related to the nature of biomass materials itself. Different biomass materials will produce AC with different surface area and pore development. The performance of OPKS-AC produced in this study was evaluated by conducting adsorption test on methylene blue.

Adsorption of Methylene Blue

Effect of adsorbent dosage. The adsorption test of methylene blue by OPKS-AC was conducted using 0.2–0.8 g litre⁻¹ of OPKS-AC dosage mixed with 25 ml of 500 mg litre⁻¹ initial concentration (C₀) of methylene blue at pH 7 for 24 hr contact time. The results of this test are shown in *Figure 3*. It is clearly observed that the percentage removal of methylene blue was increased by increasing the OPKS-AC

TABLE 2. PORE CHARACTERISTICS OF OIL PALM KERNEL SHELL-ACTIVATED CARBON (OPKS-AC) IN COMPARISON WITH COMMERCIAL AC AND COMMERCIAL AC STANDARD LIMIT

Sample	S _{BET} (m ² g ⁻¹)	S _{MICRO} (m ² g ⁻¹)	V _{MICRO} (cm ³ g ⁻¹)	V _{TOTAL} (cm ³ g ⁻¹)
OPKS-AC (This study)	305.67	112.89	0.09	0.14
*Commercial AC	541.57	223.60	0.12	0.25
Standard limit for commercial AC	300-1 500	100.1-720.0	-	0.10-0.69

Note: *Commercial AC was purchased from Sigma Aldrich in powder form.

TABLE 3. PRODUCTION OF ACTIVATED CARBON (AC) FROM VARIOUS TYPES OF BIOMASS

Biomass	Activating agent	Activation temperature (°C)	Surface area (m ² g ⁻¹)	Yield (%)	References
Oil palm kernel shell	Steam	800	584	22.0	Hidayu and Muda (2016)
Coconut shell	Steam	800	1011	24.0	Hidayu and Muda (2016)
Olive stones	Steam	750	807	25.0	Ghouma <i>et al.</i> (2015)
Date palm tree	CO ₂	576	385	28.4	Shoaib and Al-Swaidan (2015)
Yellow mombin fruit stones	KOH	500	222	12.5	Brito <i>et al.</i> (2017)
Rice husk	KOH	850	1 499-2 696	16.5-17.4	Muniandy <i>et al.</i> (2014)
Oil palm kernel shell	Steam	700	306	31.0	This study

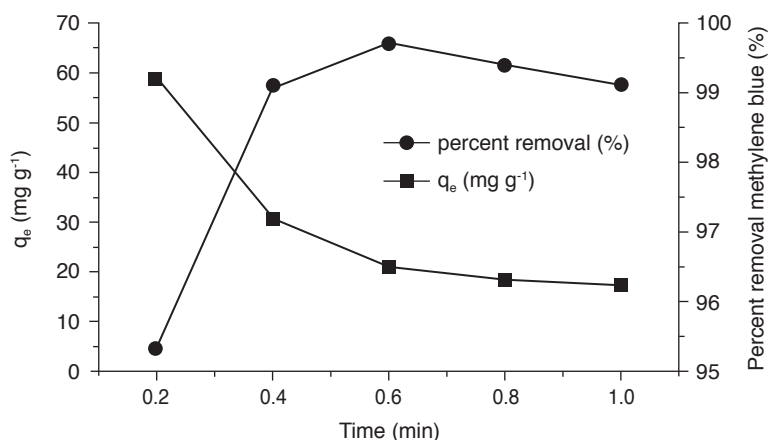


Figure 3. Effect of oil palm kernel shell-activated carbon (OPKS-AC) dosage on the removal of methylene blue and adsorption capacity.

dosage. The percentage of methylene blue removal achieved up to 99.7% when OPKS-AC dosage was at 0.6 g litre⁻¹ due to the number of available adsorption site was increased when the adsorbent dosage has been increased until it reached the stationary adsorption state (Garg *et al.*, 2003). However, increasing the adsorbent dosage can contribute to the decrease of the adsorption capacity due to the adsorption reaction remains unsaturated (Sharma and Foster, 1993). The adsorption process and adsorption capacity are dependence on the specific surface area of the materials. The adsorption process and capacity will increase when the adsorbents have wide surface area, small particle size and porous structure (Diaz *et al.*, 2013). The percentage of methylene blue removal was reduced to 99.3% when OPKS-AC dosage of 0.8 g litre⁻¹ was used.

Adsorption isotherms. The OPKS-AC produced was tested by adsorption isotherm in order to identify the best fitting isotherm for the adsorption mechanism. The adsorption isotherm studies were conducted by changing C_o of methylene blue with a fixed OPKS-AC dosage at 0.6 g litre⁻¹. The equilibrium data were analysed using Langmuir, Freundlich and Temkin equilibrium models and shown graphically in Figure 4. All of the plots show a straight line indicating that the adsorption of methylene blue is suitable with all the isotherm equilibrium models. However, the coefficients indicate that the Freundlich isotherm fitted more precisely (R²= 0.992) than the Temkin isotherm (R²= 0.867) and the Langmuir isotherm (R²= 0.826) as shown in Table 4. Freundlich model has the maximum adsorption value of 10.93 mg g⁻¹ as well as the high R² value for methylene blue adsorption on the OPKS-AC thus supporting multi-layer adsorption and physisorption mechanism. This adsorption isotherm also can be used on a heterogeneous surface energy system if the binding sites are not similar (Freundlich, 1906). Moreover, the value of n>1 represents favourable adsorption condition (Namasivayam *et al.*, 1994). This study shows that the OPKS-AC produced through carbonisation-activation system has the capacity to be used as adsorbent after being tested on methylene blue.

CONCLUSION

The carbonisation-activation system is capable to produce OPKS-AC with high fixed carbon content of 88.6%, high yield of 32.0%, and at a low activation temperature of 700°C using steam. The OPKS-AC also contains BET surface area of 305.67 m² g⁻¹, which is in the range of commercial AC standard limit. The OPKS-AC produced also has been tested on methylene blue adsorption test, which was fitted to all the isotherm equilibrium models with Freundlich model showing the best correlation

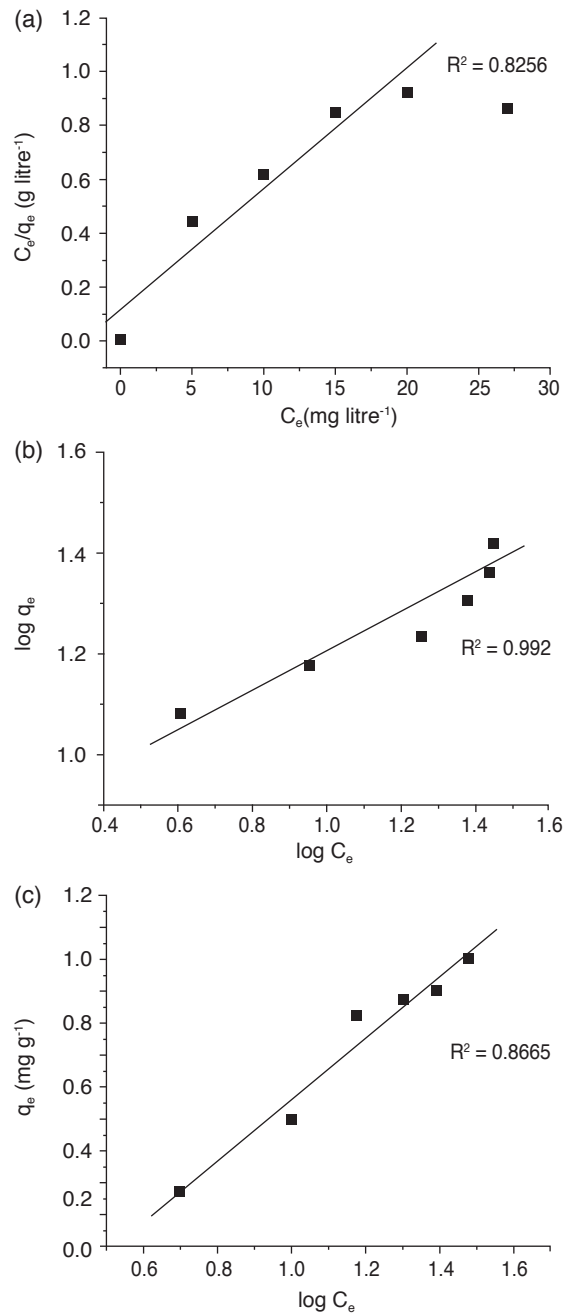


Figure 4. Langmuir (a), Freundlich (b) and Temkin (c) adsorption isotherms of methylene blue by oil palm kernel shell-activated carbon (OPKS-AC).

TABLE 4. EQUILIBRIUM MODEL PARAMETERS FOR ADSORPTION OF METHYLENE BLUE BY OIL PALM KERNEL SHELL-ACTIVATED CARBON (OPKS-AC)

Equilibrium model	Parameter	Value
Langmuir isotherm	q_m (mg g ⁻¹)	9.255
	b (litre mg ⁻¹)	54.347
	R^2	0.826
Freundlich isotherm	k_f (mg g ⁻¹) (litre g ⁻¹) ^{1/n}	10.927
	n	15.873
	R^2	0.992
Temkin isotherm	k_t (litre mg ⁻¹)	7 390.95
	b (J mol ⁻¹)	887.352
	B (litre g ⁻¹)	2.792
	R^2	0.867

coefficient of $R^2=0.992$. This study suggests that the production of OPKS-AC using carbonisation-activation system could give a great positive impact towards the environment and economic viability especially in the palm oil industry.

ACKNOWLEDGEMENT

The authors are thankful to MPOB and Universiti Putra Malaysia (UPM) for the financial support that was provided to conduct this study.

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