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Microwave irradiated coconut shell-activated carbon for decolourisation of palm oil mill effluent (POME)

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Abstract

The study was based on the adsorption principle of colour removal from POME using natural bio-sorbents Coconut Shell activated carbon (CS-AC) pre-treated by microwave irradiation. The effect of operating parameters such as pH, adsorbent dosage, and contact time was varied at different ranges: 2-8.5; 1-6 g and 1-8 hrs, respectively. It was observed that colour adsorption increased with increase in contact time. The maximum uptake of colour by the adsorbents occurred at pH 2. Also, the maximum percentage of colour removal of 95% was obtained at the optimum conditions for coconut shell-microwave (CS -MW) of 5 g/100 mL dosage, 5 hrs contact time and pH 2. The Freundlich and Langmuir isotherm linear model showed a strong correlation with the experimental data with an R^2 value of 0.9175 and 0.9892 for each of the respective model. This implies that the Langmuir isotherm model gave a better fit as indicated by the R^2 value. It can be concluded that the CSAC has a potential to be used as bio-sorbent for POME decolourization.

1. Introduction

The development of palm oil processing industry in Malaysia has led the country to become one of the largest exporters of palm oil produce in the world (Din, 2017). According to Malaysian Palm Oil Board (MPOB), as of December 2012, there were 429 palm oil processing industries which have fascinated several economic values such as national revenue, industrialization and job opportunities. However, the intense industrial palm oil processing and extraction have also resulted in the generation of a large quantity of palm oil mill effluent (POME) into the environment (Sumathi et al., 2008). Also, a report has shown that the generated POME is loaded with organic pollutants such as chemical oxygen demand (COD) and biochemical oxygen demand (BOD) (Embrandiri et al., 2015). Therefore, discharging such waste into waterways without appropriate treatment have a negative impact on the ecosystem of the aquatic life's (Hashim et al., 2017). Though, considerable efforts have been placed towards mitigating the adverse effect by reducing the pollutants concentration using different treatment approaches such as membrane technology (Subramaniam et al., 2017; Tan

et al., 2017) microbial fuel cells (Baranitharan et al., 2015; Tee et al., 2016; Neoh et al., 2017) decomposition by oxidation process (Saeed et al., 2015; Parthasarathy et al., 2016), coagulation and adsorption process using activated carbon (Mohammed and Chong, 2014; Othman et al., 2014; Alkhatib et al., 2015; Tabassum et al., 2015).

Noticeably, among the aforementioned treatments approaches, adsorption process using activated carbon have been reported most efficient, less demanding in terms of operation and cost-effective for removing the carcinogenic dark brownish colour substances such as the phenolic and melanoidin compounds present in POME (Demirbas, 2008; Ghaedi et al., 2012; Kismir and Aroguz, 2011). Adsorption treatment method for POME decolourization using activated carbon has received considerable efforts which yielded significant achievements in the last two decades. Mohammed and Chong (2014) use palm kernel shell activated carbon to decolourize POME under variable contact time, pH and adsorbent dosage. The authors reported that pH has a significant effect on decolourization performance and that almost 100% colour removal was archived at a pH

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value of 2. Equally, Alkhatib *et al*. (2015) optimize colour removal from POME using granular activated carbon and 89.95% decolourization was archived. Essentially, both studies indicated that reducing the pH level of the treating sample has a significant effect on the rate at which the adsorbate (colour compound) adheres to the active site of the activated carbon. In another application, Azmi and Yunos (2012) and Amosa *et al*. (2016) investigated the performance of coconut shell activated carbon to alleviate turbidity and suspended solids from POME. From the respective studies, 4.50 NTU, 71.26% and 89.1%, 91.4% removal efficiencies were reported. Furthermore, Adeleke *et al*. (2017) optimize removal of COD and NH_3-N from POME using powdered cow bone activated carbon. At optimal condition of pH 10, 125 g/L dosage and 2 hrs contact time, a significant COD and NH_3-N removal efficiency were observed with 89.60% and 75.61%, respectively. Recently, bio-sorbent developed from oil palm residue was used to relegate the COD and suspended solids concentration with 70.38% and 95.38% efficiency at 150 rpm agitation for a period of 24 hrs contact time (Ibrahim *et al*., 2017). In recaps, from the above literature, it is obvious that the adsorption treatment of POME by activated carbon is not only suitable for colour removal but also have the capacity to adsorb other contaminants. Though, it is a worthy note that the adsorption performance has a strong correlation with the procedures and methods used for the carbonization and activation of the char (coconut shell) which could invariably influence the nature of the final morphology of the resulted activated carbon (Tan *et al*., 2008; Basta *et al*., 2009).

Most of the activated carbon used in the studies reported above was prepared using conventional methods (physical or chemical activation) for carbonization and activation of the carbonaceous precursor. This method is demanding in terms of temperature requirement, excessive energy consumption and longer activating time (Gautam *et al*., 2014). While, the chemical method involves impregnation of the carbonaceous coconut shell with dehydrating chemical reagents such as H_2SO_4, KOH, NaOH, $ZnCl_2$, K_2CO_3 or H_3PO_4 to carbonize and activate the precursor at lower activation temperature, time and energy consumption. Compared to the former, the better porous structure is usually obtained using the chemical method of activation. However, the major limitation of this method is the need to recover the activating agent and also the generated liquid waste may require pretreatment before discharge (Yang *et al*., 2010).

Interestingly, the applications of microwave irradiation in the production and regeneration of activated carbon have grown considerably. Basically, in the microwave irradiation, interior heat is generated as a result of the dipole rotation and ionic conduction of the char particles under the application of a high-frequency voltage (Basta *et al*., 2009; Hesas *et al*., 2015). The applied field or potential voltage induces a change in particles orientation thereby synchronizing its agitation which amounted to the interior heat (Saucier *et al*., 2015). More importantly, this interior-heat significantly facilitates heat transfer and uniform distribution, hence, redeeming energy consumption and char activation period. Nevertheless, microwave irradiation heating has been applied in the preparation (Guo *et al*., 2008) and modifications (Coss and Cha, 2000; Ania *et al*., 2004; Nabais *et al*., 2004; Ania *et al*., 2005) of activated carbon. Even though, the essential literature on the significant effect on colour adsorption performance remains very scarce. In view of this highlight, microwave irradiation was used to pretreat chemically activated coconut shell and utilized for POME decolourisation at selected treatment factors which include pH, adsorbent dosage and contact time. The surface chemistry of the microwave pretreated CS-MW and non-pretreated CS-raw was characterized using the pH at the point of zero charges. In addition, the surface morphology and elemental analysis of the absorbents were examined using the Scanning Electron Microscopy (SEM) and Energy Dispersive X-ray (EDX). The equilibrium data of the adsorption process were evaluated by fitting to Freundlich and Langmuir isotherms models to determine the adsorption mechanism of the pollutant molecules.

2. Materials and methods

2.1 Preparation of POME and activated carbon

About 20 L of final discharged POME was collected from a palm oil mill at Dengkil, Selangor, filtered to remove all the visible debris and stored in a chiller at 4°C temperature. A powdered chemically activated (KOH) coconut shell carbon (CS-AC) was purchased from Merck, and this was regarded as CS-raw in this study. The CS-raw was washed thoroughly to attain a neutral pH value and also to get rid of all external impurities such as residual ash and remnant of the active reagent. Thereafter, the cleaned CS-raw was oven dried at 110°C for a period of 5 hrs and sieved to obtained particles sizes between 0.5-1.0 mm using a sieve shaker. Finally, CS-raw was subjected to pretreatment using microwave heating at a low power of 100 W for a duration of 5 mins and the resulted microwave pretreated activated carbon was stored in an airtight container and labelled CS-MW.

2.2 Characterization of activated carbon

2.2.1 Surface chemistry characterization

The pH at the point of Zero Charge was used to

characterize the surface chemistry of the CS-raw and CS-MW. Nine samples of variable pH (1-9) were prepared by a 0.05M aqueous solution of NaOH or HNO_3, while $NaNO_3$ of 0.01M concentration was applied as the electrolyte at the background (Song *et al.*, 2010). Then, 20 mL of the prepared solutions were collected into a conical flask and 0.1 g of the absorbents was added to each of the samples. In all, a total of 18 samples were prepared and placed in a swing agitator at 130 rpm and average room temperature of 298K for 48 hrs. Afterwards, the carbon was filtered, and the equilibrium pH of the samples was measured.

2.2.2 CS-AC morphology and elemental analysis

Scanning electron microscopy (SEM) (Hitachi S-3400N) Scanning Electron Microscope) was used to identify the surface physical morphology. The Brunauer-Emmett-Teller (BET) surface area and pore volume of the adsorbents were measured by multi-point BET from N_2 adsorption isotherms, using Automated Gas Sorption System (Quanta chrome Instruments).

2.3 Analytical method

The chemical parameters such as colour, chemical oxygen demand (COD), ammonia (NH_3) total phosphorus (TP), and pH of the POME were determined using standard procedure (APHA., 2005) as presented in Table 1. The colour determination was carried out by following the platinum-cobalt standard method 8025 at a wavelength of 890 nm using HACH spectrophotometer (DR 4000U). The COD, NH_3, and TP were determined using appropriate reagents. The pH value of the POME was determined using a digital pH meter.

Table 1. Physicochemical properties of the POME before and after adsorption at best condition

Parameter	Unit	DOE standard*	Before adsorption	After adsorption
Color, C_o	ADMI	100	260	13
COD	mg/L	1000	1264	972
pH	---	4-9	8.5	2.0
TSS	mg/L	200	1540	523
Ammonia	mg/L	20	3273	2634

* Source: Din (2017)

The effects of the three variables were investigated; pH, adsorbent dosages (AD) and contact time (CT). The pH was varied up to 5 different levels (2, 3, 4, 6, and 8.5), AD 6 different levels (1, 2, 3, 4, 5, 6 g) and CT was varied to 8 different levels (1, 2, 3, 4, 5, 6, 7 and 8 hrs). In each of the treatment equal volume of 100 mL sample (POME) was used under constant agitation of 50 rpm. At the end of each treatment, the final colour was determined using a HACH spectrophotometer (DR 4000U). The amount of colour removal was determined using equation 1 for each of the corresponding adsorption treatment.

$$Color\ Removal\ (\%) = \frac{(C_o - C_e)}{C_o} \times 100\ \% \qquad (1)$$

Where C_o (ADMI) is the initial concentration of colour and C_e (ADMI) is the residual colour concentration

2.4 Adsorption Isotherm

Adsorption isotherm was used to quantify the amount of adsorbate (colour removed) on the adsorbent as a function of colour concentration at a constant temperature. The application is based on the normalizing the quantity of colour adsorbed by the mass of the adsorbent and this ensures comparison with Isotherm models. In this study, Langmuir and Freundlich Isotherm Models were applied to the experimental data.

2.4.1 Langmuir adsorption isotherm

The Langmuir model assumes that uptake of colour occurs on a homogenous surface by monolayer adsorption without any interaction between adsorbed materials. Essentially, the greater values of R^2 indicate the relevance of the adsorption model for pollutants removal in POME. The value of R_L between 0 and 1 indicates that the isotherm is favourable. The isotherm is unfavorable if $R_L > 1$, linear if $R_L = 1$, irreversible if $R_L = 0$ and favorable if R_L is between 0 to 1. Mathematically, the Langmuir isotherm model is represented in equation 2:

$$\frac{C_e}{q_e} = \frac{1}{Q_o}C_e + \frac{1}{Q_o\,b} \qquad (2)$$

$$R_L = \frac{1}{1+bC_o} \qquad (3)$$

Where C_e refers to POME colour at equilibrium (ADMI); q_e (mg/g) is the amount of adsorbed pollutants; Q_o (mg/g) is the adsorption capacity related to complete monolayer coverage; b (L/g) Langmuir constant; C_o is the POME initial colour (ADMI); and b and Q_o were obtained from the slope and intercept respectively.

2.4.2 Freundlich adsorption isotherm

Freundlich isotherm model was used to consider the non-uniform distribution on the absorbent surface, which is the heterogeneity in the adsorption process. The extent of correlation between solution concentration and adsorption depends on the adsorption intensity, n. The adsorption can be linear (n = 1), chemical (n < 1), and favorable physical process (n > 1). The n and K_F are Freundlich constant and they were obtained from the graph (Figure 4(b)) as the slope and intersection respectively. Mathematically, the Freundlich isotherm model is represented in equation 4

$$log\ q_e = log\ K_F + \frac{1}{n}log\ C_e \qquad (4)$$

3. Results and discussion

3.1 Initial POME characterization

Table 1 shows the initial physicochemical characteristic of POME. The results obtained exceeded the standard limits of Department of Environment Malaysia (DOE). The results revealed that the discharged POME contain 3273 mg/L of ammonia and 1264 mg/L of COD which far above the standard limits. Also, colour content with a concentration of 260 ADMI was observed as against the standard limit of 100 mg/L and also the qualities of the POME after adsorption at best treatment conditions were as displayed in Table 1.

3.2 Characterization of microwave irradiated activated carbon

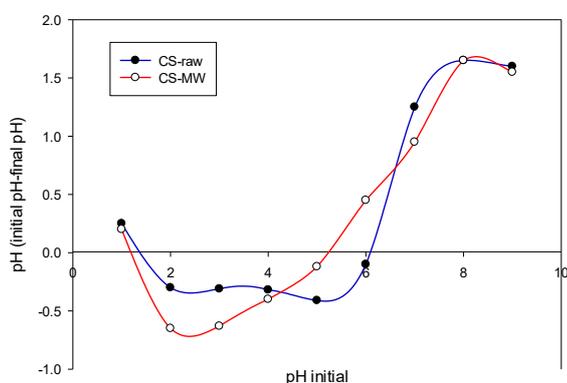### 3.2.1 pH of the point of zero charge (pH_{pzc})

Figure 1. pH at point of zero charges for CS-raw (blue curve) and CS-MW (red curve)

The point of zero charges was determined to characterize the surface chemistry of the microwave pretreated (CS-MW) and non-pretreated (CS-raw) activated carbon. Based on Figure 1, the pH_{pzc} at the point where the change in pH (initial pH-final pH) equal to 0 for the CS-MW and CS-raw where 5.3 and 6.2, respectively. More so, it is observed that the surface of the pretreated CS-MW is most negatively charged at pH 2.3 with the intensity of (-0.73) while the non-pretreated was at 5.5 with (-0.43) intensity, as presented in Figure 1. This implies that the precursor CS-raw had higher pH_{pzc} compare to the pretreated CS-MW. Essentially, the decrease in the pH_{pzc} indicates an increase in the acidic oxygen functional groups on the surface of the CS-MW (Song *et al.*, 2010). Based on the previous research, the decrease in the pH_{pzc} is as a result of the presence of the carboxyl, phenolic and carbonyl functional group on the surface of the adsorbent (Song *et al.*, 2010; Li *et al.*, 2011; Foo and Hameed, 2012). They further confirmed that these functional groups comprise the source of surface acidity. In other words, It can be deduced that the precursor (CS-raw) has a higher amount of basic sites, hence exhibit basic characteristic by adsorbing H^+ from the solution. Whereas, the pretreated CS-MW with acidic characteristic and lower pH_{pzc} readily releases H^+ to form

net negatively charged surface. This feature promotes swift adsorption of the adsorbate ions or colour from the sample.

3.2.2 Scanning electron microscopy (SEM)

SEM compared the physical changes on the surface of absorbents before and after microwave pretreatment. Figure 2(a) and (b) presents the surface morphology of the non-pretreated CS-raw and pretreated CS-MW, respectively. In Figure 2(a), a rougher surface with fewer cleavages and pores were observed. However, cleavages and pore size distribution are very important physical characteristic for the adsorption process. Also, good or uniform distribution of pores sizes which may include micro-pores, mesopores and macro-pores expedite sorption process (Ghani *et al.*, 2017). As can be seen in Figure 2(b), more gravy surfaces with a high degree of pores of variable sizes are prominent. This can be attributed to the microwave irradiation treatment of the adsorbent which resulted in creating an enabling platform for effectual removal of adsorbate (Ghani *et al.*, 2017).
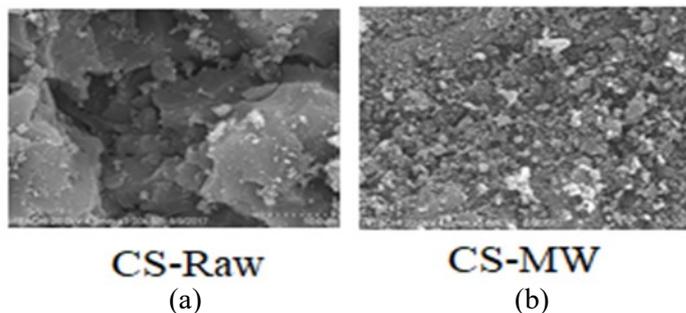

Figure 2. SEM image of the (a) non-pretreated coconut shell (CS-raw) and, (b) microwave irradiated coconut shell (CS-MW) activated carbon at X3000 magnification

The microwave at 100W increased the interior temperature of the precursor to a quasi-stationary level due to the induced change in the particles orientation and agitation (Ahmed, 2016). The author further reported that the generated heat gradually increased the interior temperature of the precursor to a quasi-stationary level. This method of heat generation ensures uniform distribution within the precursor and activation process, thus expediting the releasing of the volatile constituted substances such as the CO_2 and CO. This phenomenon increases the acidic oxygen-containing groups on the surface of the CS-MW. Morphologically, the releasing of the volatile matter accounted for the surface metamorphosis (Ghani *et al.*, 2017), as well as the creation of more active sites (carbon). This process amounted to the formation of more uniformly distributed micropores, as depicted in Figure 2. This observation was further confirmed from the results of the BET surface analysis, as presented in Table 3. From Table 3, a

considerable increase in the surface area and pores volume in the pretreated CS-MW. The specific surface area and the pore volume were observed increased by 11.88376% and 11.12867% respectively, after the microwave pretreatment. The improvement in the BET surface area was due to the oxidization effect which resulted in the releasing of volatile constituted substances, thus the formation uniform distributed pore structures on the surface (Liu *et al*., 2009; Zheng *et al*., 2014; Faraji and Ani, 2015).

3.2.3 Energy dispersive X-ray (EDX)

The EDX test conducted reveal the elemental constitutions and distribution both in the pretreated CS-MW and non-pretreated CS-raw activated carbon. Table 2 present the elemental analysis of the non-pretreated CS-raw and pretreated CS-MW activated carbon. The elements of untreated CS-raw include C, O, Al, Si and, S, while the element observed in the microwave pretreated CS-MW were C, O, Si and S. From Table 2, it is obvious that Al element completely disappeared after the microwave pretreatment, and also O, Si and S weight composition reduces significantly. This indicates that chemical reactions took place during the pretreatment. Precisely, the oxidation process greatly influences the availability of C active sites, as well as the structural reformation of the adsorbent (Wang *et al*., 2006; Liu *et al*., 2010). This implies that the oxidation process of the carbonaceous precursor accounted for the reduction in the weight composition of the trace elements (Si and S), and also the formation of more acidic oxygen-containing functional groups on the surface (Song *et al*., 2010; Ghani *et al*., 2017). These processes play a key role in the formation of more active sites for efficient adsorption process.

Table 2. Elemental analysis of activated carbon

Activated carbon	Elemental analysis (wt%)				
	C	O	Al	Si	S
CS-raw	84.42	8.56	0.39	4.81	1.82
Pretreated CS-MW	91.56	4.57	-	3.49	0.38

3.2.4 BET surface area and pores volume analysis

BET surface area and pores volume analysis for both CS-MW and CS-raw activated carbon were conducted using N_2 adsorption with Micrometric 3Flex Version 1.02 model and the detailed results are presented in Table 3. Based on Table 3, the surface area of the CS-raw was 562.5786 m^2/g while the CS-MW activated carbon was determined to be 629.4341 m^2/g. This shows that the BET surface area increased by 11.88376% after the microwave pretreatment. Similarly, a considerable increment was observed in the total pore volume with 11.12867% and 9.231679% for the adsorption and

desorption after the microwave pretreatment, respectively. In addition, the average pore diameters show a noticeable change after the microwave irradiation pretreatment, as substantiated in Table 3.

Table 3. Summary of the BET analysis for both activated carbon: pretreated (CS-MW) and non-pretreated (CS-raw)

Parameters	CS-MW	CS-raw	% increment
BET Surface Are (m^2/g)	629.4341	562.5786	11.88376
Adsorption total pore volume (cm^3/g)	0.850101	0.764970	11.12867
Desorption total pore volume (cm^3/g)	0.850101	0.778255	9.231679
Adsorption average pore diameter (Å)	51.3208	48.6132	5.569681
Desorption average pore diameter (Å)	51.3208	49.4574	3.767687

3.3 Effect of pH and contact time (CT) on decolourisation of POME using microwave irradiated activated carbon (pretreated CSAC)

Figure 3(a) illustrates the profile of pH effect and contact time on the percentage removal of colour. Principally, microwave irradiated CS activated carbons exhibited amphoteric features under variable pH which directly influence the surface positivity or negativity of the adsorbent particles (Laine *et al.,* 1989; Tan *et al*., 2008 Lim *et al*., 2010; Cazetta *et al*., 2011). The pH value was varied from 8.5 to 2 and at higher value (basic pH), anionic functional group become prominent and not suitable for the adsorption of negatively charged color causative molecules due to interionic repulsion between similarly charged particles (Mohammed and Chong, 2014; Pathania *et al*., 2017) As can be seen in Figure 3 (a), adsorption of colour increases with decrease in pH value and this phenomenon is associated not only with the negative charge on the surface of the adsorbent but also with excess OH− ions in the solution that compete for the adsorption sites. This observation was in agreement with the previous findings on the use of activated carbon to decolourize POME (Ahmad *et al*., 2005; Mohammed and Chong, 2014; Alkhatib *et al*., 2015). It is obvious that the color removal percentage increased from 78% to 95% with a decrease in pH values from 8.5 to 2. In addition, the decrease in pH caused a significant enhancement of the adsorption capacity of colour. Also, decolourisation rate increases progressively with increase in CT (Mohammed and Chong, 2014), but steady rate of color removal was observed after 5 hrs of contact. From Figure 3(a) the maximum colour removal at pH 2 was observed at 5 hrs CT while the highest
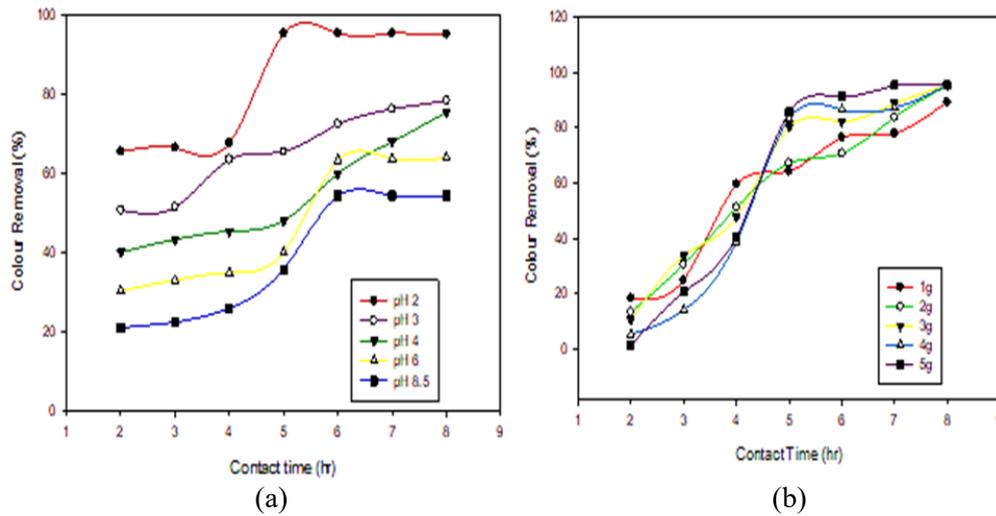
Figure 3. POME decolourisation under variable contact time at different (a) pH, and (b) dosage
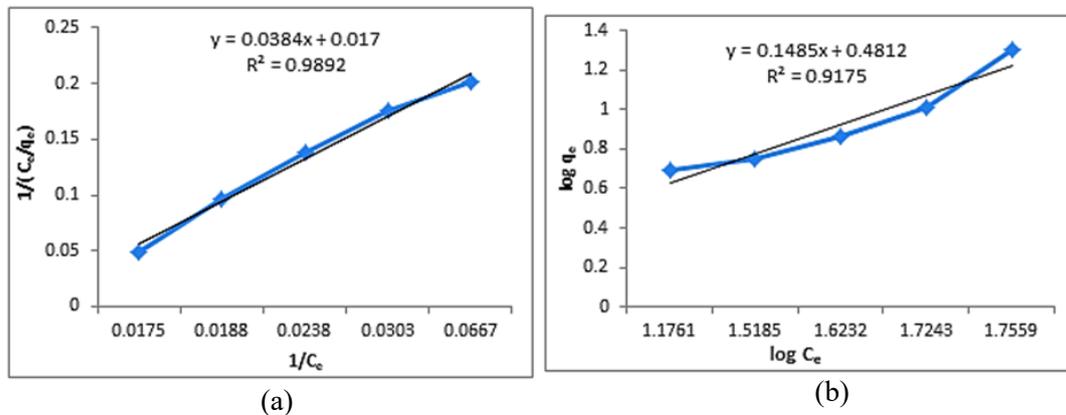

Figure 4. Adsorption Isotherm plot of (a) Langmuir; *1/(C_e/q_e) vs 1/C_e* and (b) Freundlich; *log q_e vs log C_e*

decolourisation at pH 8.5 was obtained after 7hr CT.

Effect of adsorbent dosage (AD) and CT on decolourisation using microwave irradiated pretreated CS-MW

Figure 3 (b) presents the effect of variable dosage of CS-MW at varied CT. The highest colour removal was obtained at an AD of 5 g/100 mL with 95% decolourisation. It was observed that the percentage of colour removal at equilibrium state increased from 89% to 95% when AD increases from 1 to 5 g. The results clearly indicate that the removal efficiency increases to an optimum value at an adsorbent dosage of 5 g above which further increase in adsorbent dosage has no significant effect on it. The trend of these results is in agreement with the previous findings (Gobi and Vadivelu, 2013; Alkhatib *et al*., 2015). This could be explained by the fact that the adsorption equilibrium has been reached. Also, the percentage of colour removal increased with increase in CT for all the adsorbent dosage 1 g to 6 g. As the CT increase from 1 hr to 8 hrs, the percentage of colour removal increases. The maximum percentage of colour removal for 5 g of CS-MW occurs at 7 hrs.

3.4 Adsorption isotherm

The equilibrium colour removal and adsorption characterization were examined using Langmuir and Freundlich Isotherm model. A straight line obtained from *log q_e* vs *log C_e* plot gives the slope (1/n) values of 0.148, which indicated their favourable adsorption using pretreated CSAC at the experimental conditions, as shown in Figure 4(b). The strong correlation was observed between the experimental data and the Langmuir isotherm model with R^2 of 0.989 (Figure 4(a)). This implies that the adsorbed colour was homogeneously distributed on the pretreated CSAC surface as per the assumption made for the application of the Langmuir equation, which confirms the homogeneity of the CS-MW (Alkhatib *et al*., 2015; Pathania *et al*., 2017). Also, a very good correlation was obtained between the experimental data and Freundlich model with R^2 of 0.917. Though, the coefficient of determinant archived with the Langmuir isotherm was slightly higher. In overall, the results obtained are satisfactory because of the strong agreements between the experimental data and the models. This finding is in an agreement with the previous study reported by Alkhatib *et al*. (2015).

4. Conclusion

In this work, Coconut shell-Activated Carbon was pre-treated with the microwave irradiation and applied to decolourize POME under the varied effect of pH, AD and CT. POME decolourisation is strongly influenced by pH, AD and CT. The decolourisation increases with a decrease in pH and increase in CT. Though, after the optimal CT of 5 hrs, a steady rate of decolourisation was observed at increasing AD. The optimum condition for best decolourisation of 95% was found at pH of 2, 5 g AD and 5 hrs CT. In addition, the Langmuir Isotherm Model was found favourable with an R^2 value of 0.989. Therefore, the microwave irradiation pre-treated adsorbent is expected to be feasible for the colour removal from Palm Oil Mill Effluent.

Acknowledgement

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