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Treated Coal Bottom Ash for Palm Oil Mill Effluent (POME) Decolourisation

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ABSTRACT: *The coal bottom ash (CBA) sourced from thermal power plant was chemically treated using hydrochloric acid (HCl), namely CBA-HCl, and used as an adsorbent for palm oil mill effluent (POME) decolourisation. The treated CBA-HCl was characterised for surface property via Brunauer-Emmett-Teller (BET), surface chemistry via Fourier transform infrared (FTIR) and surface morphology via scanning electron microscopy (SEM). Its effectiveness for adsorption of POME colour was investigated via batch experiments as a function of pH, initial POME concentration, adsorbent dosage and contact time. The adsorption increased with increasing contact time until an equilibrium state was achieved within 18 h to 24 h. The maximum POME colour removal, 81.15% was achieved at pH 6 utilising 14% (w/v) adsorbent dosage for 24 h. The colour adsorption isotherm and kinetics of the CBA-HCl was well-fitted with Freundlich and pseudo-second order models with correlation coefficient, $R^2 = 0.9991$ and $R^2 = 0.9996$, respectively. Based on the results, CBA can be a potential adsorbent in removing POME colour before being discharged into a watercourse.*

Keywords: Palm oil, wastewater, colour, removal, adsorption, bottom ash

1. INTRODUCTION

Palm oil is one of the major agro-industries in Malaysia covering millions of hectares of cultivated area. It has contributed substantially to economic growth and an improved living standard. Along the supply chain in the midstream sector where palm oil is extracted, a considerable amount of palm oil mill effluent (POME) is generated in mills.[1] While about 40 million tonnes of POME was reported in 2004, the figure increased to 60 million tonnes in 2009, i.e., an almost 50% increase of POME volume in 5 years.[2,3] Generally, POME is treated in a series of ponding system which involves low capital and operating cost.[4] Being polluting naturally due to high organic strength, it is also highly coloured causing photosynthesis activities reduction, and the polluted water endangers aquatic life.[5] The colour is derived from plant constituents such as tannin, lignin, humic and fulvic acid like substances, phenolic compounds as well as re-polymerisation of the colouring compound.[6] To date, the colour of POME has attracted considerable attention due to its "stubborn" characteristic and appearance as a critical water quality parameter. It affects mostly the environment which is essential to tourism. Several biological, physical and chemical methods have been proposed for POME decolourisation including membrane technology, filtration, biodegradation, oxidation, coagulation and photocatalysis.[7–12] However, many of these technologies are costly especially when applied for large scale treatment. Adsorption method, on the other hand, appears to be the most potential in wastewater treatment as it is low cost, simple and effective.[13,14]

Activated carbon has been identified as an effective adsorbent for removal of a wide variety of organic and inorganic pollutants from aqueous or gaseous media.[15] However, its application is still limited due to high cost and difficulty in regeneration.[16] This situation has driven researchers to look for low-cost adsorbent in replacing activated carbon. Several studies have revealed some low-cost adsorbents for colour removal of POME such as natural clay, palm kernel shell, fly ash and empty fruit bunch.[17–20]

Coal bottom ash (CBA) is a waste material generated abundantly from combustion of solid fuels. Its disposal at surrounding land is undesirable, and of concern since this kind of dumping is wasteful and considered highly unsuitable for agricultural utilisation.[21] The CBA appears as granules of dark grey black colour and it releases relatively less heavy metals than its fly ash counterpart.[22,23] It is also a highly porous material suitable for pollutant adsorption process.[24] It has potential for POME decolourisation based on its colour removal potential reported in the literature, owning to its good adsorption capacity by the present trace elements such as silicone and aluminium.[25–29]

This study aimed at investigating the effectiveness of CBA derived from thermal power plant for POME decolourisation. The initial POME concentration, contact time, pH and chemically-treated CBA dosage on colour removal of POME were examined. The respective colour adsorption kinetics and isotherms were also studied.

2. EXPERIMENTAL

2.1 CBA Preparation

CBA was obtained from a local coal-fired power plant in Manjung, Perak, Malaysia. The CBA obtained was a sand-like material, non-combustible and in dark grey granular form. It was soaked in distilled water (DW) at CBA:DW ratio of 1.0:1.5 for 48 h without stirring in order to remove any impurities present. The CBA was then filtered and dried in an oven at 70°C for 2 days. Next, it was ground by mortar until fairly fine and sieved to obtain CBA of equal size of 500 μm. Then, 50 g of CBA was stirred with 500 ml of 0.1 N HCl for 24 h in a beaker using magnetic stirrer.[30] The solution was left overnight to allow full sedimentation of CBA at the bottom of the beaker. The treated CBA, namely CBA-HCl was washed with an excess of DW until pH neutral. Lastly, the adsorbent was filtered and dried in an oven at 70°C for 2 days, and kept in a container prior to use.

2.2 Collection of POME

POME was collected from POME discharging pipe at a palm oil mill in Maran, Pahang, Malaysia and stored in a chiller at 4°C prior to analysis and experiment. The final POME discharge had a colour concentration of 1099 PtCo unit. It was alkaline at pH 8.4.

2.3 Adsorption Study

Adsorption experiments were performed in batches at room temperature using an orbital shaker at 160 rpm for the following operating conditions: (a) pH 5, 6, 7, 8 and 9; (b) initial POME concentrations 20%, 40%, 60%, 80% and 100%; (c) adsorbent dosages 1.8 g, 2.4 g, 3.0 g, 3.6 g and 4.2 g; and (d) contact times 0.5 h, 1 h, 1.5 h, 2 h, 3 h, 4 h, 6 h, 10 h, 12 h, 18 h and 24 h. For pH operating conditions, five 30 ml POME samples were prepared, and their pH adjusted using 0.1 N HCl or 0.1 N NaOH to the desired pH values. Next, 4.2 g of CBA-HCl was added to each sample and shaken for 24 h. The POME samples were diluted with DW to the desired concentrations. The raw POME sample representing original concentration without dilution was denoted by 100%. The pH of all samples was adjusted to pH 6. Similarly, 30 ml of each POME sample was mixed with 4.2 g CBA-HCl and agitated for 24 h. For adsorbent dosage, the desired CBA-HCl dosages of 1.8 g, 2.4 g, 3.0 g, 3.6 g and 4.2 g were added in 30 ml of each POME sample at pH 6 in a 250 ml Erlenmeyer flask. The samples were shaken at 160 rpm for 24 h. For contact time, the same experiment was repeated for different contact times using 4.2 g CBA-HCl at pH 6. Prior to colour analysis using spectrophotometer, all samples were filtered to prevent interference from adsorbent fines.

2.4 Analyses

The colour of POME was analysed using HACH spectrophotometer DR2700 at wavelength 455 nm according to platinum-cobalt standard method 8025. Scanning electron microscopy or SEM (Hitachi, S-3400N) was used to observe the morphology of CBA-HCl, and its functional groups were determined using Fourier transform infrared (FTIR) spectroscopy (Perkin Elmer Spectro 100 series USA) in the 600–4000 cm^{-1} region. Specific surface area was determined based on Brunauer-Emmett-Teller (BET) gas adsorption method using BET-Autosorb-1C (AXIS-MP-LP, Quantachrome instrument).

3. RESULTS AND DISCUSSION

3.1 CBA Characterisation

Figure 1 shows surface methodology of CBA-HCl analysed using SEM. Generally, porosity of an adsorbent has specific impact on colour adsorption. Based on the SEM images, the acid-treated CBA had highly porous structure with many small and large pores. The CBA-HCl possessed macroporous structure with pore size ranges starting from 10 μm as shown in Figure 1(b). The adsorbent surface was shiny, clean, rough and free of dust. Based on the porous structure shown, it was concluded that HCl treatment had managed to increase the surface area and charges of CBA which would have positive influence during colour adsorption of POME.

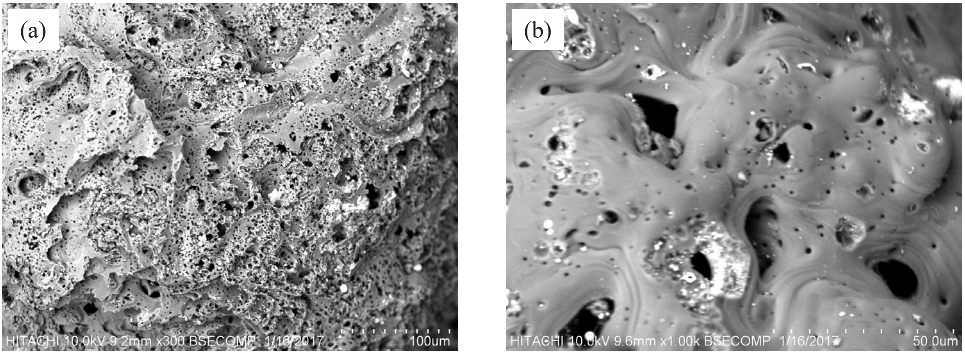

Figure 1: Surface morphology of CBA-HCl at (a) 300X magnification, and (b) 1000X magnification.

3.1.1 BET surface area analysis

The BET surface analysis was conducted to study the characteristics of the sample pores. Based on Tables 1 and 2, the untreated CBA had higher surface area (6.80 $m^2\ g^{-1}$) compared with other untreated CBA obtained elsewhere. The CBA-HCl in this study exhibited a 6-fold surface area increment, i.e., 44.15 $m^2\ g^{-1}$ after acid treatment, which was indicative of a good activation employed. The higher the surface area of CBA-HCl, the more surface areas are available for adsorption; thus providing greater adsorptive capacity during POME colour adsorption.[31,32]

Table 1: BET analysis for untreated and treated CBA in this study.

Sample	BET surface area ($m^2\ g^{-1}$)	Pore volume ($cm^3\ g^{-1}$)	Pore width (Å)
Untreated CBA	6.80	0.1151	677.09
CBA-HCl	44.15	0.1535	139.02

Table 2: Specific surface area of bottom ash from various sources.

Sample	BET surface area ($m^2\ g^{-1}$)	Source	Reference
Untreated CBA	6.80	Manjung, Perak	Present study
CBA-HCl	44.15	Manjung, Perak	Present study
Untreated CBA	1.04	Kapar, Selangor	Present study
Untreated CBA	0.17–1.00	NA	Jayaranjan et al.[33]
Untreated CBA	1.25	Shanxi, China	Sun et al.[34]
Untreated CBA	1.77	Corlu, Turkish	Dincer et al.[23]

Notes: NA denotes no information available

3.1.2 FTIR analysis

The various functional groups present on the surface of the CBA-HCl were identified by FTIR spectroscopy (Figure 2). It was found that CBA had a strong presence at 3746 cm^{-1} which was characteristic of a non-acidic Si-OH group. Its presence was originated from -OH of water molecules associated with silica-rich extra-framework debris.[35,36] Broad band at around 3501 cm^{-1} was assigned to hydroxyl (-OH) groups and amine groups.[37] The O-H stretching vibrations occurring within a broad band indicated presence of "free" -OH groups as well as bonded O-H groups of carboxylic acids.[38] Meanwhile, the bands at 787 cm^{-1} represented SiO-H vibration.[39] The FTIR spectrum showed the presence of many polar functional, i.e., carboxyl, carbonyl, hydroxyl and amino groups which were responsible and involved in colour binding during POME treatment using CBA-HCl.[38]

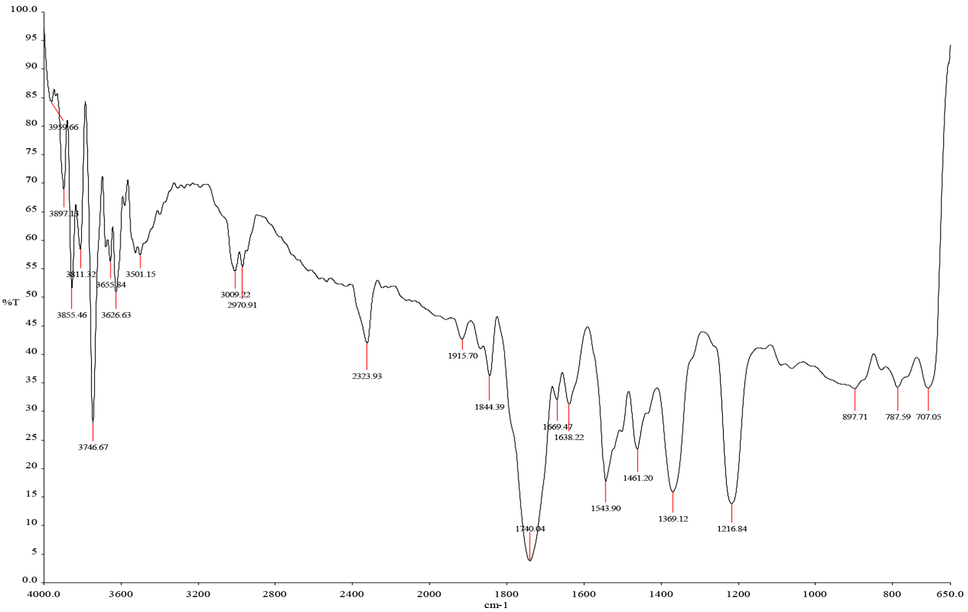

Figure 2: FTIR spectrum of CBA-HCl.

3.2 Adsorption Analysis

3.2.1 pH of POME

Figure 3 shows the effect of pH on percentage removal of colour from POME. The pH of a solution plays a vital role in adsorption process as it influences the sensitivity of functional groups of adsorbate.[20] According to Mohammed, changes in solution pH affect adsorbent surface load and influence the degree of colour compounds attraction.[20] In this study, pH 6 gave the highest percentage of colour removal, i.e., 81.15% among the pH used as a result of higher degree of protonation of the active sites on CBA-HCl surface. At this pH, the surface of adsorbent was positively charged and this facilitated sorption of anioinic functional groups of coloured compounds which led to better POME colour removal.[20] As pH increased or decreased, positively charged sites reduced, hence lower POME colour removal as shown by lower adsorption efficiency of CBA-HCl, i.e., 77.92%, 73.16%, 71.97% and 70.18%, at pH 5, 7, 8 and 9, respectively. The findings indicated that pH indeed had a significant effect on the performance of CBA-HCl for POME decolourisation.

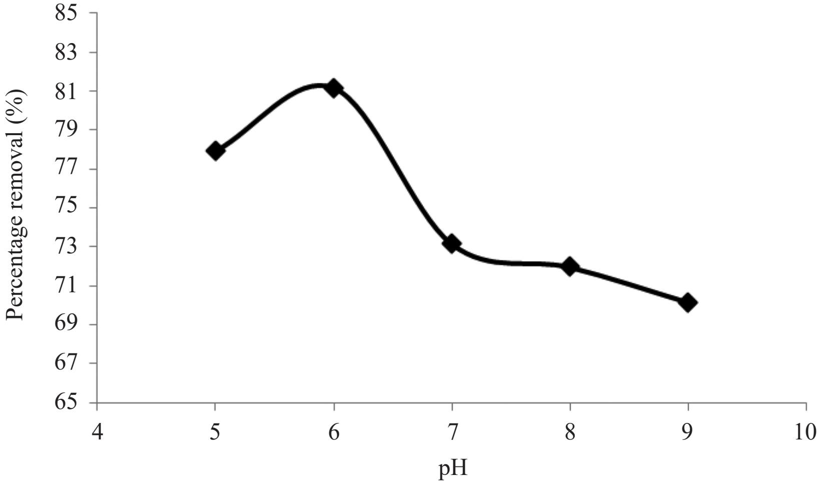

Figure 3: Effect of pH on percentage colour removal of POME.

3.2.2 Adsorbent dosage

The CBA-HCl dosage was varied from 6% to 14% (m/v). Figure 4 shows the effect of adsorbent dosage on percentage removal of POME colour. The percentage of POME colour removal increased with increasing of CBA-HCl dosage. Adsorption rate depends on driving power per unit area.[20] As dosage of adsorbent increased, more surface area would be provided to adsorb colour from POME. In Figure 4, colour removal from POME was most efficient at the highest CBA-HCl dosage of 14% (w/v) due to the highest number of available adsorption sites. The higher the adsorbent dosage, the higher the availability of adsorption sites which can enhance the performance of colour removal. However, the percentage of removal does not increase proportionately with the adsorbent dose. Although significant colour removal was observed at lower dosage but beyond 10% (m/v), the increase had only gone up slightly by about 3%.

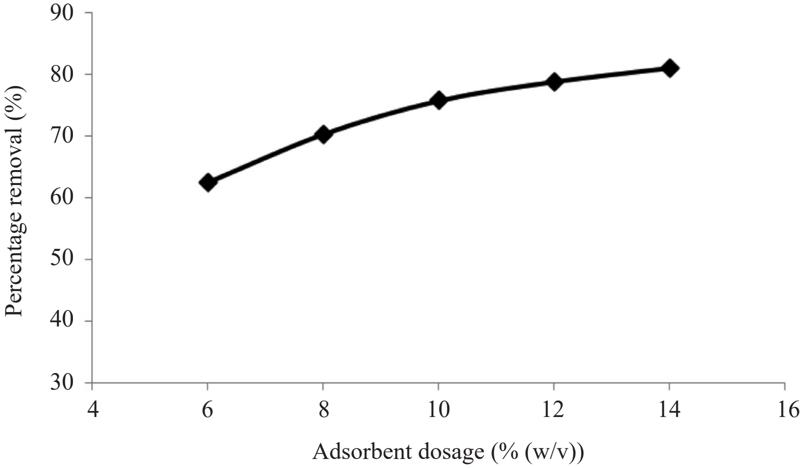

Figure 4: Effect of adsorbent dosage on percentage colour removal of POME.

3.2.3 Contact time

The effect of contact time was studied to determine the sorption rate of POME colour onto CBA-HCl and its associated equilibrium time. Figure 5 shows that the adsorption of POME colour reached equilibrium within 24 h. Concentration of POME colour decreased drastically in the first 3 h and remained constant from 18 h to 24 h. The optimum contact time was found at 18 h for adsorption of POME colour. The adsorption rate was rapid initially due to greater availability of actives sites on CBA-HCl surface. As time passed, more and more active sites got occupied by coloured molecules resulting in much saturated surface and lesser available sites to be offered for adsorption. This phenomenon created a repulsive force between the coloured molecules in medium and on CBA-HCl surface, hence the percentage colour removal of POME gradually slowed down.[40]

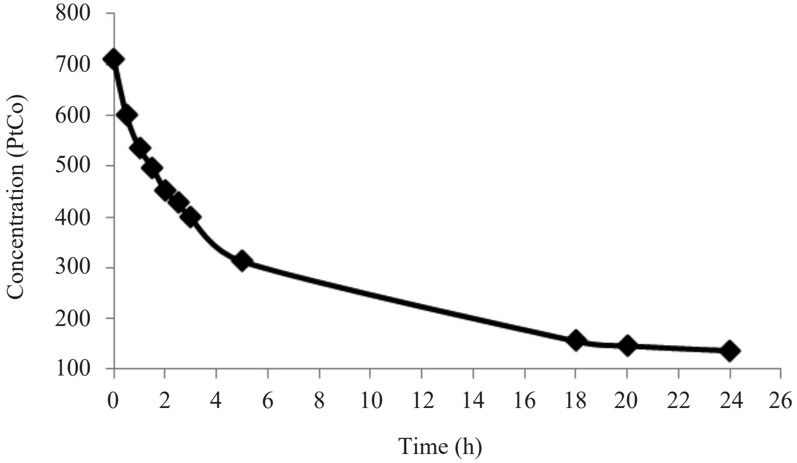

Figure 5: Effect of contact time on colour removal of POME.

3.2.4 Adsorption capacity

Adsorption capability of CBA-HCl on POME colour was shown in Figure 6. The adsorption capacity (q_e) increases linearly with increasing final concentration of POME. The maximum q_e of CBA-HCl was recorded at 5.70 PtCo/g. This q_e was way lower than those of other adsorbents as presented in Table 3. Some precipitates had formed after the adsorption experiment probably due to chemical reaction between trace elements present in CBA-HCl and some reactive POME components.

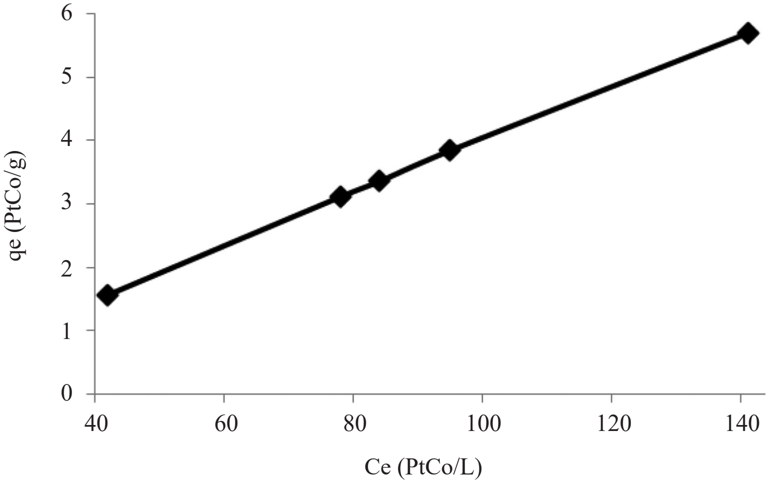

Figure 6: Equilibrium adsorption of POME colour at various initial concentration.

Table 3: Comparison of adsorption capacity (q_e) of CBA-HCl with other studies.

Adsorbents	q_e (PtCo/g)	References
CBA-HCl	5.70	Present study
Coconut shell activated carbon (CSAC)	39.10	Sia et al.[41]
Palm kernel shell activated carbon	227.27	Jalani et al.[18]
Montmorillonite	1.00	Said et al.[17]
Microwave activated palm kernel shell (PKSM)	191.00	Mohammed[20]
Microwave activated empty fruit bunch (EFBM)	186.50	

3.3 Adsorption Isotherm

It is important to establish the most appropriate correlation for the equilibrium curves in order to optimise the design of an adsorption system for adsorption of colour from POME. Langmuir and Freundlich models were used to describe the equilibrium characteristics of POME colour adsorption. The Freundlich equations deployed as in Equation 1 was linearised as shown in Equation 2. As for Langmuir, the linearised equation is given by Equation 3.

$$q_e = k_f C_e^{1/n} \tag{1}$$

$$log\ q_e = 1/n\ log\ C_\mathrm{e} + log\ k_f \tag{2}$$

$$C_e / q_e = 1/q_\mathrm{m}\ C_\mathrm{e} + 1/(k_L q_\mathrm{m}) \tag{3}$$

In the equations, q_e is the amount of pollutant adsorbed per gram of adsorbent at equilibrium (mg g^{-1}), C_e was the equilibrium concentration of pollutant in the solution (mg l^{-1}), and k_f and n are the constants for Freundlich to indicate the q_e and intensity, respectively. These constant values can be obtained from the slope and intercept of the plot of log q_e versus log C_e while q_m and k_L are the Langmuir constants that can be obtained from the slope and intercept from the plots of C_e/q_e versus C_e. The q_m value is the maximum pollutant uptake capacity for the respective adsorbent and conditions. In this case, unit of C_e is expressed as PtCo/L.

The best-fitted POME colour adsorption onto CBA-HCl with Freundlich isotherm ($R^2 = 0.9991$) reveals a heterogenous surface binding mechanism for adsorption of colour onto CBA-HCl compared to the less fitted Langmuir isotherm ($R^2 = 0.6194$) as shown in Figure 7. Meanwhile, Table 4 lists the respective constant values used.

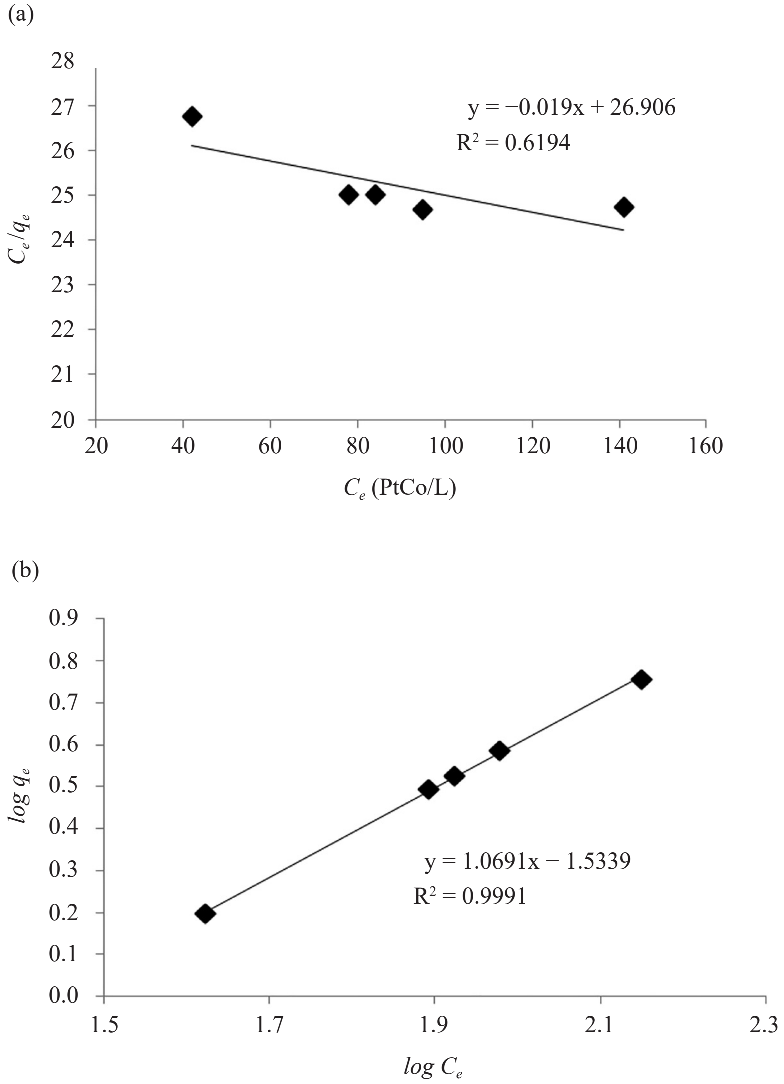

Figure 7: Isotherm model of (a) Langmuir, and (b) Freundlich for POME colour adsorption by CBA-HCl, respectively.

Table 4: Adsorption isotherms parameters.

	Langmuir isotherm			Freundlich isotherm		
Parameter	q_{max} (mg g^{-1})	K_L	R^2	K_F	n	R^2
Colour	–52.6316	0.00071	0.6194	0.0292	0.9354	0.9991

3.4 Adsorption Kinetics

The experimental adsorbate uptake rates onto CBA-HCl was investigated using kinetic models. The adsorption kinetic based on the overall adsorption rate of CBA-HCl was analysed by pseudo-first order and pseudo-second order models as shown in Figure 8. The R^2 value of pseudo-second order, shown in Figure 8(b), was higher compared with R^2 value of pseudo-first order, as in Figure 8(a). Thus, the pseudo-second order model was most suitable to describe the adsorption kinetic involved. The findings showed that the step controlling adsorption rate of CBA-HCl was chemical sorption and POME colour adsorption rate was affected by the number of available adsorption sites on the surface of CBA-HCl rather than POME solution concentration.[41,42] Table 5 shows the respective q_e, K_1 of first order adsorption, and K_2 of pseudo second order adsorption.

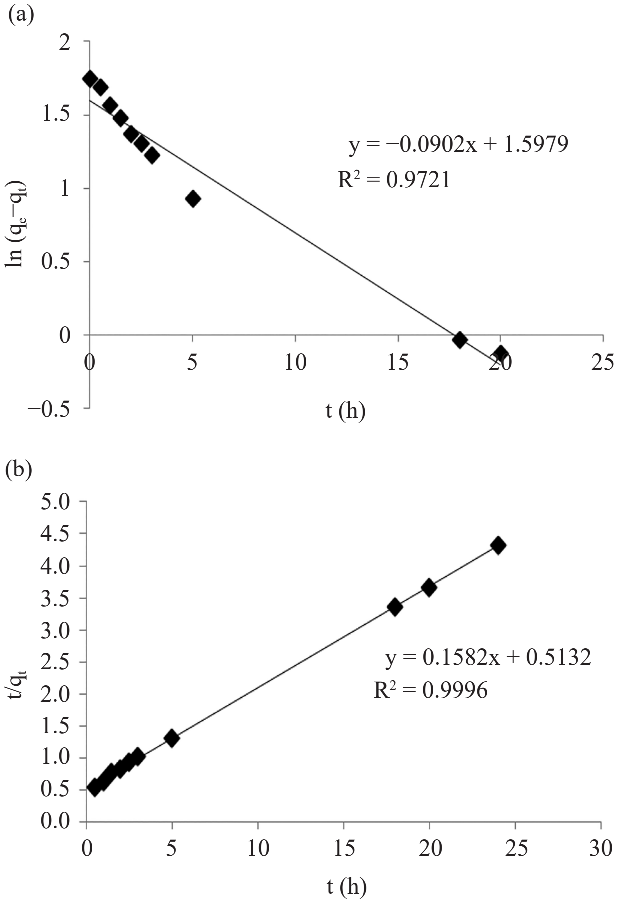

Figure 8: Kinetic model of (a) pseudo-first order, and (b) pseudo-second order for POME colour adsorption by CBA-HCl, respectively.

Table 5: Adsorption kinetic parameters.

	Pseudo-first order			Pseudo-second order		
Parameter	q_e (mg g^{-1})	K_1 (1 h^{-1})	R^2	q_e (mg g^{-1})	K_2 (g mg^{-1} h^{-1})	R^2
Colour	4.9426	0.0902	0.9721	6.3211	0.0488	0.9996

4. CONCLUSION

A by-product of thermal power plant, namely CBA, had ability to remove coloured compounds of POME. The treated CBA-HCl had developed more pores and bigger surface area after acid treatment. This material performed well in colour removal of POME, hence showing potential for colour adsorption during POME treatment. The optimum contact time, CBA-HCl dosage and pH for POME colour removal were 18 h, 10% (w/v) and pH 6, respectively. It was found that Freundlich and pseudo-second order models were best fitted for POME colour adsorption. The acid-treated CBA can be an alternative adsorbent in wastewater treatment.

5. ACKNOWLEDGEMENTS

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