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## Removal of phenol by activated carbons prepared from palm oil mill effluent sludge

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**Abstract:** The study was attempted to produce activated carbons from palm oil mill effluent (POME) sludge. The adsorption capacity of the activated carbons produced was evaluated in aqueous solution of phenol. Two types of activation were followed, namely, thermal activation at 300, 500 and 800°C, and physical activation at 150°C (boiling treatment). A control (raw POME sludge) was used to compare the adsorption capacity of the activated carbons produced. The results indicated that the activation temperature of 800°C showed maximum absorption capacity by the activated carbon (POME 800) in aqueous solution of phenol. Batch adsorption studies showed an equilibrium time of 6 h for the activated carbon of POME 800. It was observed that the adsorption capacity was higher at lower values of pH (2–3) and higher value of initial concentration of phenol (200–300 mg/L). The equilibrium data were fitted by the Langmuir and Freundlich adsorption isotherms. The adsorption of phenol onto the activated carbon POME 800 was studied in terms of pseudo- first and second order kinetics to predict the rate constant and equilibrium capacity with the effect of initial phenol concentrations. The rate of adsorption was found to be better correlation for the pseudo-second order kinetics compared to the first order kinetics.

**Keywords:** activated carbon; adsorption; palm oil mill effluent (POME) sludge; phenol

### Introduction

Phenolic compounds are released in wastewater of many industries during their manufacturing processes such as paint synthetic rubber, pharmaceuticals, oil and gasoline, paper and pulp, and wood preserving chemicals (Aksu and Yener, 1998; Aleksieva *et al.*, 2002; Kim *et al.*, 2002; Jung *et al.*, 2001; Rao and Viraraghavan, 2002). Phenol is a high-priority persistent, hazardous, bioaccumulative and toxic chemical once released to the environment may increasing long-term toxic effects to human health and the environment, even if the release was of a small amount (Carlson, 1999). Because of their toxicity, phenols have been included in the list of priority pollutants by Malaysian Government (ILBS, 1998). Removal or biodegradation/bioconversion of phenol from wastewater is therefore necessary before it is discharged to receiving water bodies.

Phenol removal from wastewaters by activated carbon adsorption method is an alternative. Activated carbon adsorption is a well-established, powerful and most widely used method for treating domestic and industrial effluents since it has large adsorption capacity (Aksu and Yener, 1998; Calace *et al.*, 2002; Gupta *et al.*, 2000; Chen *et al.*, 2002), fast adsorption kinetics and relative ease of regeneration (Jung *et al.*, 2001; Viraraghavan and Alfaro, 1998). But the cost for preparing commercial activated carbon is quite expensive.

One promising and comparatively new approach is the production of cheaper activated carbons from agro-industrial waste such as palm oil mill effluent

(POME) sludge as the biosolids for the potential applications of phenols adsorption. POME is a mixture of high-polluted effluent (from sterilizer and oil room) and low polluted effluent (steam condensate, cooling water, boiler discharge and sanitary effluent), which effects are contributed in serious environmental problems through the pollution of water bodies (Ma, 2000). In Malaysia, very limited researches have addressed on the production of activated carbons from oil palm (OP) industrial waste although OP industries generate 10 million tons of POME every year (Deraman, 1993; MPOB, 2003). Currently several treatment processes have been practiced to treat POME. The most commonly used is the biological treatment of POME using anaerobic treatment systems. Although this system needs only simple construction and operation, the anaerobic pond requires large land area (COD volumetric loading between 1 to 5 kg/(m<sup>3</sup>·d)) with limited treatment efficiency of the system (limit up to 60%–70% of BOD<sub>5</sub> removal in order to maintain adequate nutrient ratio for further aerobic treatment required for river discharge) and regular desludging is also required. These processes are not sufficient to handling and disposal concern in terms of discharge standard regulation, environmental protection and economic viability (Ma, 2000; Ahmad *et al.*, 2002; Rusnani *et al.*, 1999). Therefore the aim of this study was to produce activated carbon using thermal and physical activation utilizing the POME as abundant raw material for the application of phenol adsorption.

### 1 Materials and methods

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1.1 Sample collection

POME sludge was collected from palm oil industry (Seri Ulu Langat Palm Oil Mill Sdn. Bhd.) in Dengkil, Selangor, Malaysia. POME sludge was in

liquid form pored in the plastic container and stored in the cold room at 4°C for further use. The characteristic of POME sludge is shown in Table 1.

Table 1 Characteristics of palm oil mill effluent (Ma, 2000)

Parameters	Concentration	Elements	Concentration
pH	4.7	Phosphorus, mg/L	180
Oil and grease, mg/L	4000	Potassium, mg/L	2270
Biochemical oxygen demand, mg/L	25000	Magnesium, mg/L	615
Chemical oxygen demand, mg/L	50000	Calcium, mg/L	439
Total solids, mg/L	40500	Boron, mg/L	7.6
Suspended solids, mg/L	18000	Iron, mg/L	46.5
Total volatile solids, mg/L	34000	Manganese, mg/L	2.0
Ammoniacal nitrogen, mg/L	35	Copper, mg/L	0.89
Total nitrogen, mg/L	750	Zinc, mg/L	2.3

1.2 Pre-treatment of sample and production of activated carbon

POME sludge was available in liquid form with suspended solids. The samples were washed successively 2 to 3 times with distilled water to remove residual oil and other suspended materials. Then, the samples were filtered and dried (105°C) to remove the moisture content and stored at 4°C until the process for activated carbon production.

Two types of activation: thermal and physical activation were conducted to produce activated carbon. For thermal activation, the processed sample was heated in the high temperature chamber furnace (CARBOLITE, UK) at 300, 500 and 800°C for 30 min followed by crushing and sieving to size fraction less than 125 µm. During physical activation, the raw material was boiled in distilled water at 150°C for 2 h in an oven and therefore the treated sample was dried at 105°C until constant weight. The dried sample were crashed and sieved to the 125 µm. The activated carbons were designed as the POME C for inactivated (control, raw POME), POME B for boiling treatment, and POME 300, POME 500 and POME 800 for the thermal treatment at 300, 500 and 800°C, respectively. All activated carbon were stored at 4°C until further use for adsorption test.

1.3 Batch adsorption test

The adsorbents (activated carbon) prepared from POME sludge were studied for the removal efficiency of phenol from aqueous solution under different experimental conditions. 0.25 g of activated carbon was added into 100 ml conical flask containing 25 ml aqueous solution of phenol at pH 3 with concentration of 50 mg/L. The samples were agitated in a rotary shaker at room temperature (30 ± 2°C) for 24 h at 150 r/min. At 1, 2, 3, 4, 5, 6, 12, 18 and 24 h of interval, samples (5 samples: physical activated 1, thermal

activated 3, and control-untreated sample 1) were with- drawn to determine the residual concentration of phenol and its equilibrium time. The activated carbon and solutions were then separated by filtration. The total phenol content was determined according to the Box (1983). The absorbance was measured at 725 nm against distilled water and reagent blank using a UV spectrophotometer (Anthelie Junior, SECOMAM). The final concentration was measured from the standard curve with known concentrations.

The same experimental conditions (contact time 6 h, adsorbent dose 0.25 g, agitation 150 r/min) were studied to determine the optimum pH and initial phenol concentration varying with the pH values of 2, 3, 5 and 8, and concentration of 25, 50, 100, and 200 mg/L respectively. All the data were average of three replicates.

The adsorption capacity *q* was calculated from the difference between the initial and equilibrium adsorbate (phenol) concentration which is as follows:

$$q = \frac{(C_0 - C_e)}{M} V$$

(1)

where *q* is the adsorption capacity (mg/g), *C*<sub>0</sub> and *C*<sub>e</sub> are the initial and equilibrium concentration (mg/L) respectively, *M* is the adsorbent dosage(g) and *V* is the solution volume (L).

2 Results and discussion

2.1 Production of activated carbons

Four types of activated carbon were produced from POME sludge, three (POME 300, POME 500 and POME 800) from thermal activation (300, 500 and 800°C) and one (POME B) from boiling treatment at 150°C. The color of activated carbon produced from physical activation (boiling treatment) and thermal treatment at 300°C were similar with control (inactivated), which was brown. The other two types

of activated carbon produced from thermal treatment at 500 and 800°C were black in color. However, it could be seen that activated carbon produced at 800°C, was much darker compared to others.

2.2 Phenol released in aqueous solution from activated carbons

Since phenol was being leached from the activated carbons in aqueous solution during adsorption test, the phenol released rate was determined to evaluate the actual adsorption capacity of each adsorbent. The results showed that only activated carbons produced at temperature 800°C (POME 800) released very low concentrations of phenol (<1 mg/L) into distilled water (Table 2). The concentration of phenol released were observed to the order of POME C > POME B > POME 300 > POME 500 > POME 800. The releasing rate was increased for all samples except for POME C where the concentration of phenol released decreased after 12 h. The releasing rate was higher for first 1 h of operation after that the rate was slow until 24 h of treatment for most of the cases.

Table 2 Concentration of phenol released by various activated carbons produced from POME sludge in aqueous solution

Time, h	Concentration of phenol released, mg/L				
	POME C	POME B	POME 300	POME 500	POME 800
1	136.81	117.09	41.09	29.62	0.52
2	137.29	117.27	41.29	30.13	0.66
3	137.76	117.44	41.50	30.64	0.69
4	138.23	117.62	41.70	31.15	0.74
5	138.71	117.80	41.91	31.66	0.75
6	139.18	117.98	42.12	32.18	0.81
12	142.02	119.04	43.35	35.24	0.85
18	137.14	120.11	44.58	38.30	0.87
24	132.22	121.18	45.82	41.37	0.91

2.3 Adsorption test

2.3.1 Effect of contact time

The adsorption capacity of phenol were observed decreasing in the order of POME B > POME 300 > POME 500 > POME C > POME 800(Fig.1). The concentration of phenol treated by activated carbon produced from POME at temperature 800°C (POME 800) largely decreased from initial concentration of 50 to 15.6 mg/L within 1 h of operation. The percentage removal for first hour is around 68.7% and has the adsorption capacity about 3.44 mg/g. The concentrations then continued to decrease, and percentage removal and adsorption capacity still increasing with respect to the time and reached the equilibrium point at 6 h. The activated carbons POME C, POME B, POME 300, and POME 500 were found to be removed to the 33.8%, 28.3%, 25.7% and 25.4% of phenol respectively from an initial concentration of 50 mg/L. Özkaya (2005) has studied on the commercial activated carbon for removal of phenol from aqueous

solution indicated that the absorption capacity approximately 17 mg/g was observed within two hours of contact time. In another study, Rengaraj *et al.* (2002) observed that the adsorption capacity by the palm seed coat carbon and commercial activated carbon for removal of phenol was about 19 mg/g and 9 mg/g for 3 and 6 h of contact time respectively. The results showed that all treatment had relatively low adsorption capacity compared with the control sample (without activation) except for the activated carbon POME 800. Therefore the studies on the effect of pH and initial phenol concentration were conducted with activated carbon (POME 800) produced by thermal activation at 800°C.

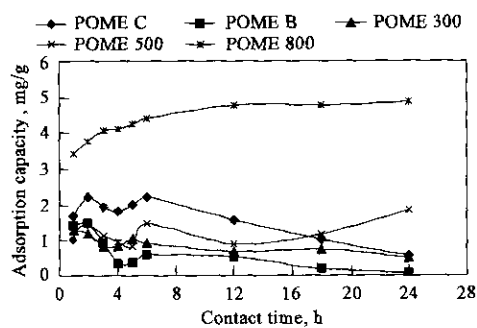


Fig.1 Adsorption capacity of activated carbon produced from POME sludge in aqueous solution of phenol  
Adsorbent dosage: 1% w/v; initial concentration: 50 mg/L; temperature: 30°C; pH: 3; agitation: 150 r/min

2.3.2 Effect of initial pH

The variation of pH on adsorption capacity of POME 800 in aqueous solution of phenol is shown Fig.2. The adsorption capacity of POME 800 was decreased from 4.23 to 4.06 mg/g when the pH of the initial phenol solution was increased from pH 2 to 5. The phenol removal was remained constant with the pH range of 2 to 3. However, adsorption capacity was increased to 4.24 mg/g as pH was increased from pH 5 to 8. It may be related to the presence of excess H<sup>+</sup> ions accelerating the removal of the phenol with the anion OH<sup>-</sup> in the aqueous solution. It is also possible that the surface properties of activated carbon are depended on the pH of the solution. The previous

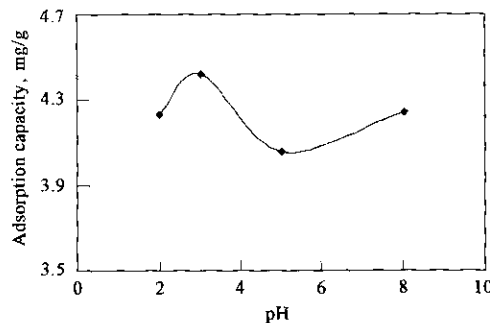


Fig.2 Removal of phenol by activated carbon POME 800 with the variation of pH  
Adsorbent dosage: 1% w/v; concentration: 50 mg/L; temperature: 30°C; agitation: 150 r/min; contact time: 6 h

study by Aksu and Yener (1998) also found the isoelectric point for activated sludge would be usually between pH 1 and 3.

### 2.3.3 Effect of initial phenol concentrations

The effect of initial phenol concentrations (25–200 mg/L) on adsorption capacity by activated carbon (POME 800) is shown in Fig.3. The adsorption capacity of POME 800 was increased from 2.61 to 10.68 mg/g with the initial values from 25 to 200 mg/L indicating that the phenol removal (55.8%–93.7%) is dependent on initial concentrations. The increase of adsorption capacity of adsorbent may be due to the higher probability of collision between phenol and adsorbent. Increasing the adsorbate concentration generally caused a decreasing in adsorption capacity. Lower initial concentration also favored higher adsorption yield which was the limit of 200 mg/L in this study. Further higher concentration (>200 mg/L) may reduce the adsorption capacity with the activated carbon (Aksu and Yener, 1998).

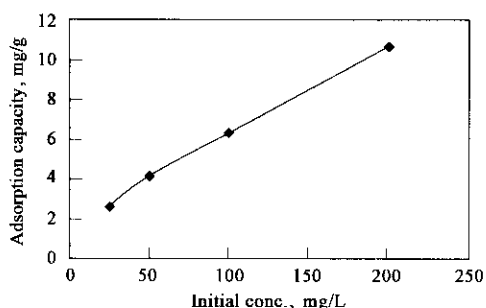


Fig.3 Effect of initial phenol concentration on adsorption by activated carbon POME 800

Adsorbent dosage: 1% w/v; temperature: 30°C; pH: 3; agitation: 150 r/min; contact time: 6 h

## 2.4 Adsorption isotherms

Equilibrium studies on adsorption are provided information on the capacity of the adsorbent or the amount required to remove a unit mass of pollutant. An adsorption isotherm is characterized by certain constant values of which express the surface properties and affinity of the adsorbent and can also be used to compare the adsorptive capacities of the adsorbent for different pollutants. Such a plot is usually fitted to either the Freundlich or Langmuir isotherms at a constant temperature. These equations are in common use for describing adsorption isotherms for water and wastewater treatment applications.

The Freundlich isotherm has been widely adopted to characterize the adsorption capacity of organic pollutants using different adsorbents by fitting the adsorption data. The Freundlich equation is used for heterogeneous surface suggesting that binding sites are not equivalent and/or independent. The Freundlich isotherm has the general form such as:

$$q_e = K_F C_e^{1/n} \quad (2)$$

This equation can be modified as:

$$q_e = \frac{C_0 - C_e}{M} = K_F C_e^{1/n} \quad (3)$$

where  $q_e$  is the adsorption capacity (mg/g),  $C_0$  and  $C_e$  are the initial and equilibrium concentration (mg/L) respectively,  $M$  is the adsorbent dose (g),  $K_F$  and  $1/n$  are the adsorption capacity and intensity of adsorption respectively. The values of  $K_F$  and  $1/n$  can be determined from the intercept and slope, respectively, of the logarithmic plot in Eq.(3).

$$\ln q_e = \ln K_F + 1/n \ln C_e \quad (4)$$

In order to facilitate the estimation of the adsorption capacities at various conditions, the Langmuir adsorption isotherm, a typical model for monolayer adsorption onto a surface with a finite number of identical sites was applied. The linearized Langmuir model can be written as:

$$C_e/q_e = \frac{1}{K_L q_m} + \frac{1}{q_m} C_e \quad (5)$$

where  $q_e$  is the amount of dye adsorbed at equilibrium (mg/g),  $C_e$  is the equilibrium concentration of phenol,  $q_m$  (mg/g) and  $K_L$  (mg/L)<sup>-1</sup> are the Langmuir constants, representing the maximum adsorption capacity for the solid phase loading and the energy constant related to the heat of adsorption. The constants  $q_m$  and  $K_F$  can be evaluated from the intercept and slope of the linear plot of the experimental data of  $C_e/q_e$  vs.  $C_e$ .

The applicability of the isotherm equations is compared by judging the correlation coefficients,  $R^2$ . These adsorption isotherms were made based on the data obtained for phenol adsorption starting at different initial concentrations. The concentrations are in the range of 25–200 mg/L. Measurements were carried out after adsorbent contacting periods equal to the equilibrium times (6 h) previously determined in the effect of contact time study.

In Freundlich, a plot of  $\ln q_e$  vs.  $\ln C_e$  enabled the empirical constant  $K_F$  and  $1/n$  to be determined (Table 3) from the intercept and slope of the linear regression is shown in Fig.4. For Langmuir, a plot of  $C_e/q_e$  vs.  $C_e$ , the  $q_m$  and  $K_L$  were determined from its slope and intercept (Fig.5) is shown in Table 3.

The correlation coefficient showed that in general, the Freundlich model ( $R^2=0.990$ ) fitted the result better than the Langmuir model ( $R^2=0.957$ ). The Freundlich model fitted the data significantly when a change in the mechanism of adsorption with concentration was considered. The Langmuir model equation is used for homogenous surfaces. The Freundlich isotherm model assumes neither homogeneous site energies nor limited levels of adsorption can result from the overlapping patterns of several Langmuir-type adsorption phenomena occurring at

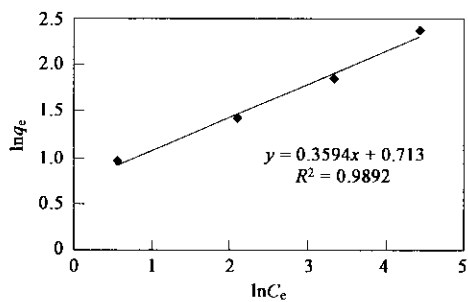


Fig.4 Linearized Freundlich adsorption isotherm of phenol  
Adsorbent dosage: 1% w/v; temperature: 30°C; pH: 3; agitation: 150 r/min

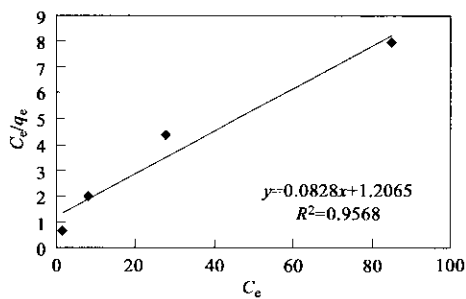


Fig.5 Linearized Langmuir adsorption isotherm of phenol  
Adsorbent dosage: 1% w/v; temperature: 30°C; pH: 3; agitation: 150 r/min

Table 3 Langmuir and Freundlich isotherms parameters for phenol adsorption by activated carbon (POME 800)

Isotherms		Parameters		
Langmuir	$q_{\text{m}}$ mg/g	$K_L$ , L/mg	$R^2$	
	12.078	0.069	0.957	
Freundlich	$K_{\text{F}}$ , (mg/g)(mg/L) <sup>-1/n</sup>	1/n	$R^2$	
	2.048	0.358	0.990	

different sites on complex adsorbents (Calace *et al.*, 2002).

In the case of sewage sludge by chemical activation (Calvo *et al.*, 2001) a good fitting was obtained by using the Langmuir model but not Freundlich model. These differences in the goodness of fit for both models considered here arise from the assumption in the case of Langmuir model of surface energetic homogeneity, an assumption not accepted in the frame of Freundlich model.

2.5 Adsorption kinetics

The adsorption kinetics are probably the most important factor for adsorber design with adsorbate residence time and the reactor dimensions controlled by the system's kinetics. However, adsorption kinetics show a large dependence on the physical and/or chemical characteristics of the adsorbent materials which also influences the adsorption mechanism. In order to gain a better understanding of the mechanism and to quantify the rates of adsorption, it was

necessary to propose the suitable model equations for the adsorption of phenol by activated carbon produced from POME sludge. For this purpose, the Lagergren (1898), a pseudo-first order mechanism and equations of Ho (1995), a pseudo-second order mechanism were used to model experimental data.

The adsorption kinetics may be described by a pseudo first order (Lagergren, 1898). The differential equation is as follows:

$$dq/dt = K_1(q_e - q_t) \tag{6}$$

Integrating Eq. (6) for the boundary conditions  $t=0$  to  $t=t$  and  $q_t=0$  to  $q_t=q_t$ , gives:

$$\log(q_e - q_t) = \log q_e - (K_1/2.303)t \tag{7}$$

which is the integrated rate law for a pseudo-first order reaction. Where  $q_e$  is the amount of phenol adsorbed at equilibrium (mg/g);  $q_t$  is the amount of phenol adsorbed at time  $t$ (mg/g) and  $K_1$  is the equilibrium rate constant ( $\text{h}^{-1}$ ). In order to obtain the rate constants the straight line plots of  $\log(q_e - q_t)$  versus  $t$  for different treatment conditions were analyzed. The rate constant ( $K_1$ ) and correlation coefficients  $R^2$  values under different conditions of phenol adsorption were calculated from these plots.

The adsorption kinetics may be described by a pseudo-second order (Ho, 1995). The differential equation is as follows:

$$dq/dt = K(q_e - q_t)^2 \tag{8}$$

Integrating Eq.(8) for the boundary conditions,  $t = 0$  to  $t = t$  and  $q_t=0$  to  $q_t = q_t$ , rearranging the final equation as:

$$t/q_t = 1/Kq_e^2 + t/q_e \tag{9}$$

which is the integrated rate law for a pseudo-second order reaction. Where  $q_e$  is the amount of phenol adsorbed at equilibrium (mg/g);  $q_t$  is the amount of phenol adsorbed at time  $t$  (mg/g) and  $K$  is the equilibrium rate constant ( $\text{g}/(\text{mg} \cdot \text{h})$ ). The straight line plots of  $t/q_t$  versus  $t$  were tested to obtain rate parameters. The values of  $K$  and  $R^2$  were calculated from the equation under different conditions of phenol adsorption.

The constant adsorption parameter  $K_1$  and  $K$  for pseudo- first and second order kinetics as well as the regression coefficients ( $R^2$ ) were determined from the plots of  $\log (q_e - q_t)$  against time  $t$ , and the plot of  $t/q_t$  against time  $t$  are presented in Fig.6 and Fig.7 respectively. The results for the constant parameters and regression coefficients are shown in Table 4. The results indicated the straight lines which showed the applicability of first-order rate expression of Lagergren and second-order rate expression of Ho respectively. The correlation coefficient showed that in general, the pseudo-second order equation of Ho ( $R^2=0.999$ ) was better fitted by the experimental

results compared to the Pseudo-first order equation of Lagergren ( $R^2=0.977$ ) for activated carbon produced from POME at activation temperature of 800°C. The constant parameter  $K_1$  in first order kinetics indicates the phenol adsorbed per gram of adsorbent for every hour of operation. The results indicated that 0.40 mg of phenol was adsorbed by each gram of activated carbon POME 800 for every hour of operations. The constant parameter  $K$  for second order kinetics equation indicates the amount of adsorbent needed in gram per unit of phenol in each hour of operations. 0.64 g of POME 800 was needed to adsorb per mg of phenol in 1 h of operation. The previous study tested by plotting  $t/q_t$  against  $t$  for sorption of heavy metals (lead-II and copper-II) from ash showed the Pseudo-second order equation is better than those in

Pseudo-first order kinetics (Kaur *et al.*, 1991).

3 Conclusions

Four types of activated carbon were produced from POME sludge: three from thermal activation (300, 500 and 800°C) and one from boiling treatment at 150°C. Among the activated carbons, the POME 800 showed good adsorption capacity with very low content of phenol released in aqueous solution. A contact time of 6 h was needed for the adsorption of phenol in equilibrium. It was observed that the adsorption capacity was higher at lower values of pH (2–3) and higher value of initial concentration of phenol (200–300 mg/L). The data were fitted well by Freundlich isotherm ( $R^2=0.990$ ) and pseudo-second order kinetic ( $R^2=0.999$ ) compared to Langmuir isotherm ( $R^2=0.957$ ) and pseudo-first order kinetic ( $R^2=0.977$ ). Overall, activated carbon POME 800 was found to be promising for the application of phenol removal in industrial wastewater reducing its production cost and to be encouraged to waste management concerning of waste utilization and disposal of POME sludge.

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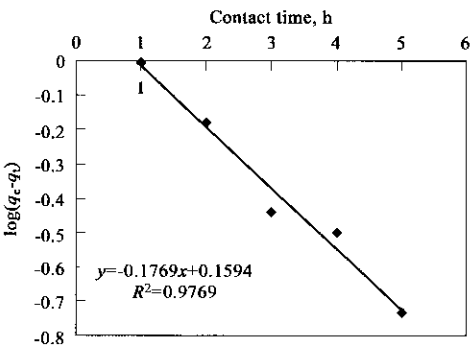


Fig.6 Pseudo-first order kinetics of phenol onto activated carbon POME 800  
Adsorbent dosage: 1% w/v; temperature: 30°C; pH: 3; agitation: 150 r/min

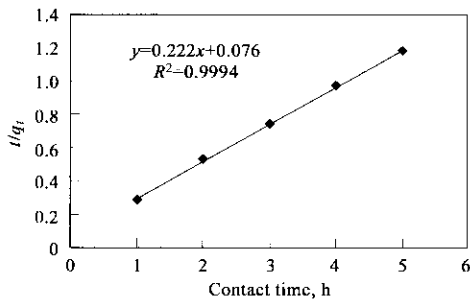


Fig.7 Pseudo-second order kinetics of phenol onto activated carbon POME 800  
Adsorbent dosage: 1% w/v; temperature: 30°C; pH: 3; agitation: 150 r/min

Table 4 Pseudo- first and second order equation parameters for adsorption of activated carbon (POME 800) in aqueous solution of phenol

Kinetics	Parameters	
Pseudo-fist order equation	$K_1, h^{-1}$	$R^2$
	0.405	0.977
Pseudo-second order equation	$K, g/(mg \cdot h)$	$R^2$
	0.644	0.999

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