

Contents lists available at [ScienceDirect](http://www.sciencedirect.com/science/journal/13858947)

Chemical Engineering Journal

journal homepage: www.elsevier.com/locate/cej

The factors affecting the performance of activated carbon prepared from oil palm empty fruit bunches for adsorption of phenol

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article info

Article history: Received 31 July 2008 Received in revised form 18 November 2008 Accepted 15 July 2009

Keywords: Oil palm empty fruit bunches Activated carbon Adsorption Phenol Statistical optimization Thermal activation

ABSTRACT

Powdered activated carbons (PACs) were produced from oil palm empty fruit bunches (EFB) by varying the operating parameters of temperatures, $CO₂$ gas flow rates and activation times using 2-level full factorial experimental design. The EFB samples were first carbonized for 30 min using nitrogen gas followed by physical activation using $CO₂$ to optimize best production conditions. The optimum conditions for PACs produced were investigated through adsorption tests on aqueous solution of phenol. The results of this study demonstrated that the activation temperature with the range of 800–900 ◦C had the most significant effect on the adsorption characteristics as well as the yield of the activated carbon produced. Based on the analysis of variance (ANOVA) and model equation developed, the optimum production conditions for the EFB PAC were found to be at the activation temperature of 900 \degree C with CO₂ gas flow rate of 0.1 L/min and activation time of 15 min. Characterization of PAC produced showed that the activation conditions would find good-quality adsorbent with the BTE surface area of 345.1 m^2/g and well forming pores distribution. © 2009 Elsevier B.V. All rights reserved.

1. Introduction

Malaysia is the largest oil palm producer in the world and generates one of the most abundant residues namely empty fruit bunches (EFB), amounting to 12.4 million tonnes annually [\[1\]. A](#page-7-0) small quantity is used as fuel for the boilers in the oil palm mills while most are unused and disposed in landfills. However, due to limited landfill sites as well as the additional treatment of the leachate from the landfill, studies of effective utilization and recovery of the EFB have become an urgent and significant issue.

The adsorption process has an edge over the other treatment methods due to its sludge free operation. Activated carbon is the most widely used adsorbent for this purpose. The wide usefulness of activated carbon is a result of its chemical and mechanical stability, high adsorption capacity and high degree of surface reactivity [\[2,3\].](#page-7-0) Extensive studies have shown that activated carbon (AC) is efficient in the adsorption of numerous bio-resistant organic pollutants from an aqueous system [\[4,5\].](#page-7-0) Activated carbon adsorption has been recommended by the USEPA as one of the best available technologies (BAT) [\[6\]](#page-7-0) in the removal of organic compounds. The major constraint of its usage in industrial applications is high production cost due to expensive materials used.

Many investigators have studied the feasibility of less expensive materials of agricultural by-products resulting from the annual harvesting and industrial processing of various agricultural crops for production of activated carbon and its application in wastewater treatment. The by-products have been shown to be potential materials in the production of activated carbons with efficient adsorptive capacity comparable with that of commercial activated carbons [\[7–15\].](#page-7-0)

Phenolic compounds are considered to be hazardous wastes, when released into the aquatic environment by industries such as coke ovens in steel plants, petroleum refineries, petrochemical, phenolic resin, pharmaceutical, chemical and dye industries, etc. [\[16–18\]. T](#page-7-0)he discharge of phenolic waste into waterways may adversely affect human health as well as that of flora and fauna. Ingestion of a small amount of phenol (5 ppm) by human beings may cause nausea, vomiting, paralysis, coma, greenish or smoky coloured urine, and even death from respiratory failure or cardiac arrests. Phenolic compounds are present in palm oil mill effluent (POME) at high concentrations (>1000 mg/L) which are generated abundantly in oil palm industries. Conversion of EFB to activated carbon which can be used in the treatment of POME for the removal of phenolic compounds is a win–win situation [\[14\]. I](#page-7-0)t is imperative to remove phenol up to regulatory standards from wastewater before being discharged into waterways.

The present work aims to demonstrate the production of powdered activated carbons derived from oil palm industrial waste namely EFB and their adsorptive capacities on phenol removal.

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^{1385-8947/\$ –} see front matter © 2009 Elsevier B.V. All rights reserved. doi:[10.1016/j.cej.2009.07.033](dx.doi.org/10.1016/j.cej.2009.07.033)

This study adds new useful value to EFB as well as handling it in safe sanitary way. The PAC produced is environmentally friendly with wide industrial applications especially in water and wastewater treatments due to its adsorption properties. Different operating parameters including temperatures, activation times and $CO₂$ gas flow rates were investigated to evaluate the performance of activated carbons produced in aqueous solution of phenol by statistical design.

2. Material and methods

2.1. Raw material and its preparation for activated carbon

The oil palm empty fruit bunch (EFB) sample was obtained from the Seri Ulu Langat Palm Oil Mill in Dengkil, Selangor, Malaysia. The EFB was collected in plastic bags and preserved in a cooling room at 4 ◦C. The EFB was washed and dried in the oven at 105 ◦C for 24 h for dehydration until a constant weight was obtained.

2.2. Production of activated carbons

Physical activation comprised the thermal treatment (carbonization) using nitrogen gas followed by activation with $CO₂$ gas at different temperatures, times and gas flow rates which was adopted to produce powder activated carbons (PACs). The range and level of the variables are shown in Table 1. It was selected based on experimental works done by other researchers [\[13,19,20\].](#page-7-0)

A 2-level full factorial design (FFD) with two central points was selected for the optimization of PACs production. The total number of experiments for the production of PACs determined by the statistical software through the design of experiment (DOE) was 10. The EFB was carbonized and activated in a high temperature horizontal furnace (CARBOLITE, UK) in which the inside temperature varied from 100 to 1200 \degree C. The EFB samples were placed in the middle of a ceramic tube of 5 and 6 cm inside and outside diameter respectively. The tube was inserted inside the horizontal furnace and its two ends were sealed by the inlet and outlet metal enclosures. The inlet enclosure contains two holes which were connected to nitrogen and $CO₂$ gas cylinders by aluminium tube system. Two flow meters, one for nitrogen gas and the other for $CO₂$ gas were fixed on an aluminium panel and connected to the system.

Nitrogen gas (N_2) was applied to the system at 2.5 L/min [\[19\]](#page-7-0) once the tube furnace had been switched on and set at required temperatures (600, 750 and 900 \degree C) and continued for 30 min with the heat rate of the horizontal furnace set at $23 °C/min$. The flow rate of nitrogen gas then was stopped while the carbon dioxide gas $CO₂$) was released and continued for the activation times of 15, 30 and 45 min. The activated carbons produced were taken out from the ceramic tube, ground and sieved to size fractions of \leq 250 $\rm \mu m$ (0.25 mm).

2.3. Adsorption test

The adsorption of phenol by PACs obtained by the FFD was studied at various contact times i.e. 15 min, 30 min, 45 min, 1 h, 2 h, 3 h, 4 h, 5 h, 6 h, 12 h, 18 h, 24 h and 48 h. 0.25 g of each powdered activated carbon produced was added into a 100 ml conical

Table 1 Experimental factors and their levels for fractional factorial design (FFD).* .

Central point: average of high and low values for each parameter.

Table 2

Operating conditions of adsorption test by the powdered activated carbons (PACs) produced.

PAC samples produced	AC1, AC2, AC3, AC4, AC5, AC6, AC7, AC8, AC9 and AC10
Contact time, h	0.25, 0.5, 0.75, 1, 2, 3, 4, 5, 6, 12, 18, 24, 48
Phenol aqueous solution	Concentration = $50 \,\mathrm{mg/L}$ pH ₆ Sample volume = 25 ml
PAC dose Agitation speed Temperature	10 g/L 150 rpm Room temperature (26 ± 1 °C)

flask containing 25 ml aqueous solution of phenol at pH 6 with a concentration of 50 mg/L. The sample was agitated at 150 rpm in a rotary shaker at room temperature (26 ± 1 °C). The detail optimization study is shown in Table 2. At the end of each interval of the time mentioned, the powdered activated carbon was separated from the treated solution by filtration using the ADVANTEC qualitative filter paper no.131. The total phenol content in the solution was determined according to the method of Box [\[21\]. T](#page-7-0)he absorbance was measured at 725 nm against distilled water and reagent blank using Optima UV spectrophotometer (model SP-3000 plus). The final phenol concentration was measured from the standard calibration curve. The removal percentage of phenol (%) and adsorption capacity (mg/g) were calculated using the following equations:

$$
Removal of phenol(\mathscr{X}) = \frac{(C_0 - C_f)}{C_0}
$$
\n(1)

Adsorption capacity,
$$
q_t = \frac{[(C_i - C_f)V]}{M}
$$
 (2)

where q_t is the adsorption capacity (mg/g) at time *t*; C_i and C_f are the initial and final phenol concentrations (mg/L) respectively. *M* is the adsorbent dosage (g) and *V* is the volume of solution (L). All the data presented are the average of the three replicates. The standard deviation (SD, \pm) for each data point is determined and shown accordingly.

2.4. Analytical methods for determination of physical properties of the EFB-based PAC produced

2.4.1. Scanning electron microscope image

The scanning electron microscopy (SEM) technique was employed to observe the surface physical morphology of the oil palm EFB derived activated carbon. The JSM-5600 model of the Japanese manufacturer (JEOL Ltd.) was used for this purpose. It has a magnification range from $15\times$ to 200,000 \times and a resolution of 5 nanometers. It is capable of producing high-resolution images of a sample surface.

2.4.2. PAC Surface measurement

A fully automated analyzer for surface area and pore size measurements of Autosorb 1 model and Quantachrom brand (USA) was used to determine the BET surface area of the best quality PAC produced. The instrument can determine the BET surface area with remarkable sensitivity.

2.5. Statistical analysis

The second order quadratic coefficients were calculated using the software Design-Expart®6.0.8 (Stat Ease Inc., Minneapolis, USA). The statistical analysis of the model was performed in the form of analysis of variance (ANOVA) which includes Fisher's *F*-test (for overall model significance), its associated probability *p*(*F*) and determination coefficient, *R*2. It also includes the Student's *t*-value for the estimated coefficients and the associated probabilities *p*(*t*).

Fig. 1. Adsorption capacity of activated carbons produced by the different experimental designs in aqueous solution of phenol with time (a) high temperature-RUN 3: 900 °C, 45 min, 0.1 L/min CO₂; RUN 4: 900 °C, 15 min, 0.1 L/min CO₂; RUN 5: 900 °C, 15 min, 0.25 L/min CO₂; RUN 8: 900 ℃, 45 min, 0.25 L/min CO₂ (b) Low temperature-RUN 1: 600 °C, 45 min, 0.25 L/min CO₂; RUN 2: 600 °C, 15 min, 0.1 L/min CO₂; RUN 7: 600 ◦C, 45 min, 0.1 L/min CO2; RUN 9: 600 ◦C, 15 min, 0.25 L/min CO2 (c) Medium temperature (central) RUN 6: 750 ℃, 30 min, 0.175 L/min CO₂; RUN 10: 750 ℃, 30 min, 0.175 L/min CO₂.

The 3D response surface and 2D contour plot were presented to evaluate the parameters tested within the surface of response.

3. Results and discussion

3.1. Adsorption capacity test for the powder activated carbon produced

The relationship between contact time and adsorption capacity of PACs in the removal of phenol is presented in Fig. 1(a–c). It is observed that the highest adsorption occurred during the initial stage of 15 min except for the PAC samples prepared at 600 ◦C, after which the performance gradually increased slightly and became almost constant. This observation is in support of the findings reported by several authors [\[18,22\].](#page-7-0)

For the PACs produced at 900 ℃ at varying activation times and $CO₂$ gas flow rates, the adsorption capacity is shown in Fig. 1(a). The results showed that 96.5% of phenol adsorption occurred at 15 min contact time where the adsorption then increased slightly with the increase in contact time before reaching equilibrium. The adsorption property of the PACs produced at high activation time together with high $CO₂$ gas flow rate was found to be lower than of the other PAC produced at lower activation time. The highest adsorption recorded was 99.5% at 24 h contact time.

A comparison of the different PACs prepared at 600 ◦C showed that only 30% of adsorption was performed within 15 min which then increased with time to reach the highest value of 92% after 48 h (Fig. 1(b)). The results also showed that a lower activation time had a significant effect on the adsorption properties of the PACs while the flow rate of $CO₂$ had little effect.

For the PACs produced at 750 \degree C at the activation time of 30 min and $CO₂$ gas flow rate of 0.175 L/min, the results are presented in Fig. 1(c). The adsorption of phenol achieved during the first 15 min was 96%. The adsorption then increased slightly with the increase in contact time and became constant. The highest adsorption recorded was 99.36% at 24 h contact time.

3.2. Optimization of physical conditions for powdered activated carbon produced

A 2-level full factorial design was used for the development of AC production to obtain the relation between preparation variables to the adsorption capacity and yield. Since in most of the cases, the equilibrium adsorption was found at the initial 15 min of contact time, the adsorption capacities within this time for all PACs prepared were considered in the determination of the optimum physical conditions (Table 3). The maximum adsorption capacity of PACs was observed from 1.03 to 4.83 mg/g, while the carbon yield obtained ranged from 19.3% to 27.9%. The regression model was developed to determine the optimum activation conditions of activated carbons for the improvement of adsorption capacity (mg/g)

Table 4

Analysis of variance (ANOVA) for selected factorial model for adsorption capacity.

and activated carbon yield (%). The model equations are as follows:

$$
Adsorption capacity (mg/g) = +2.91 + 1.58A - 0.22B - 0.18C \\
$$

$$
-0.092AB - 0.14AC - 0.23BC \quad (3)
$$

$$
Yield(\%) = +24.87 - 2.88A - 1.22B + 0.16C - 1.10AB
$$

$$
+0.17AC + 0.040BC \t\t(4)
$$

where *A* the is activation temperature in ◦C, *B* and *C* are the activation time in minutes and $CO₂$ gas flow rate in L/min respectively.

The adequacy of the models was justified through the analysis of variance (ANOVA) for the response of the PAC's adsorption capacity. In Table 4, the *F*-value of 92.70 implies that the model was significant. Values of "Prob > *F*" less than 0.05 indicated that the model terms are significant. The result also indicated that the activation temperature (*A*) was highly significant since its "Prob > *F*" value was 0.0019. Therefore, it had a major effect on the adsorption properties of the PACs produced. The "Curvature *F*-value" of 151.43 implies that there is significant curvature (as measured by difference between the average of the center points and the average of the factorial points) in the design space.

The ANOVA for the PACs yield is shown in Table 5. The model *F*-value of 428.05 implies that the overall model was significant. Furthermore, the variables *A*, *B*, *C*, AB, AC and BC were significant model terms. For the activated carbon yield, activation temperature was found to have the greatest effect with the highest *F*-value of 1936.95 as shown in Table 5. However the activation time had less effect than the activation temperature as its *F*-value in Table 5 is 286.96, while the $CO₂$ gas flow rate showed very little effect on activation with *F*-value of 13.92.

Fig. 2. Theoretical vs. experimental values of activated carbon produced (a) adsorption capacity in aqueous solution of phenol (b) yield of activated carbon.

Table 5

Analysis of variance (ANOVA) for selected factorial model for yield of activated carbon.

Fig. 3. 3D surface plots: effect of physical conditions on adsorption capacity of the PAC produced. (a) Effect of activation temperatures and times on adsorption capacities of the PAC produced. (b) Effect of activation temperatures and CO₂ gas flow rates on adsorption capacities of the PAC produced. (c) Effect of activation times and CO₂ gas flow rate on adsorption capacities of the PAC produced.

[Fig. 2\(a](#page-3-0) and b) shows the theoretical versus the experimental values for adsorption capacity and yield respectively. It is clearly shown that the theoretical values obtained were quite close to the experimental values, indicating that the models developed were successful in bridging the correlation between the activated carbon preparation variables to the adsorption capacity and yield. The quality of the model developed was evaluated based on the coefficient of determination value. The experimental values of both responses are correlated with the theoretical responses [\(Fig. 2\(a](#page-3-0) and b)). The coefficient of determination R^2 of 0.9964 and 0.9992 showed that 99.64% and 99.92% of the variations in adsorption capacity and yield respectively can be explained by the independent variables; activation temperature, activation time and $CO₂$ gas flow rate. Both the R^2 values obtained were very high, indicating that there was a good agreement between the experimental and the theoretical (predicted) values from the models. The model also showed that *R*² is in reasonable agreement with the adjusted R^2 values of 0.9857 and 0.9969 for adsorption capacity and yield respectively. The standard deviations of the two models were 0.2 and 0.17 for Eqs. [\(3\) and](#page-3-0) [\(4\)](#page-3-0) respectively. The closer the R^2 value to unity and the smaller the standard deviation, the better the model will be as it will give theoretical values closer to the experimental values for the response.

The graphical representations of the model equations facilitate an examination of the effects of PAC preparation factors on the adsorption capacity. The 3D surface and 2D contour plots between the production factors are shown in Figs. 3 and 4. These results illustrate the responses of different experimental variables and can be used to identify the major interactions between the variables [\[23\].](#page-7-0) The 3D response surface and contour plots in Fig. 3(a and b) show that the adsorption capacity of activated carbon samples increased with the increase in activation temperature of 600–900 °C. The results also showed that the adsorption capacity of PACs generally decreased with the increase in the activation time of 15–45 min and $CO₂$ gas flow rate of 0.1–0.25 L/min. The response in Fig. 3(c) shows a curvature, which indicates a good interaction effect between $CO₂$ gas flow rate 0.1–0.25 L/min and activation times of 15–45 min on adsorption of phenol by the PACs. The results clearly showed that the activation temperatures have significant effect on the adsorption properties of the PACs produced while the activation times with $CO₂$ gas flow rate had little influence. The results obtained agreed with the work by Sudaryanto et al. [\[24\]](#page-7-0) who reported that the activation time gave no significant effect on the pore structure of the activated carbon produced from cassava peel, and the pore characteristics changed significantly with the activation temperature. It

Fig. 4. 3D surface plots: effect of physical conditions on carbon yield. (a) Effect of activation temperature and activation time on carbon yield. (b) Effect of Activation temperature and CO2 gas flow rate on carbon yield. (c) Effect of activation time and CO2 gas flow rate on carbon yield.

had also been found that activation time did not show much effect on the surface area obtained for activated carbons prepared from apricot stones using steam activation [\[25\].](#page-7-0)

The interaction effect between the activation temperature, activation time and gas flow rate on the carbon yield was studied and found to have significant effects on the response (yield). Fig. 4(a–c) represents the three-dimensional responses, which were constructed to show the effect of activated carbon production variables on the carbon yield. It was found that the carbon yield decreased with increasing activation temperature, activation time

Fig. 5. Porosity images observed by Scanning Electron Microscope (a) Raw EFB, (b) selected PAC produced (RUN 4) and (c) Commercial PAC.

Table 6

The adsorption capacity of PAC produced and PAC commercial and its removal of phenol in aqueous solution.

^a 900 °C, 15 min, 0.1 L/min CO₂ gas.

b Charcoal activated carbon (HmbG Chemicals).

and $CO₂$ gas flow rate. The highest yield was obtained when all the three variables were at the minimum value within the range studied. This result agreed with the results of the works done by other researches [\[24,26\].](#page-7-0)

The statistical analysis of the Design-Expert software gave seven numerical solutions of optimum conditions with desirability values of 1.0–0.933 for the production of PACs based on maximum adsorption capacity. The optimum factors determined by numerically using software with high desirability (1.0) were as follows: activation time of 900 \degree C, activation time of 15 min and CO₂ gas flow rate of 0.1 L/min.

3.3. Scanning electron microscope images for powder activated carbons at different conditions

The scanning electron microscopy (SEM) technique was employed to observe the surface physical morphology of the PACs. [Fig. 5\(a](#page-5-0)–c) shows the SEM images of the raw powdered EFB, the selected activated carbon sample produced and commercial powdered activated carbon with $2000 \times$ magnification. It is shown in [Fig. 5\(a](#page-5-0)) that there were very little pores available on the surface of the precursor. However, after the activation process under the optimum preparation conditions of 900 ◦C activation temperature, 15 min activation time and $CO₂$ gasification at 0.1 L/min, pores of different sizes and different shapes were developed as shown in [Fig. 5\(b](#page-5-0)). Pores in [Fig. 5\(b](#page-5-0)) are comparable to that of the commercial one in [Fig. 5](#page-5-0) (c), which is observed to have lots of impurities. During physical activation by $CO₂$ gas, the diffusion of the oxidising agent through the carbonaceous matrix may have enhanced the removal of impurities and the activation of the carbon to create porosity according to the following reaction: $C + CO₂ = 2CO$. The micro-porosity is open and widened with even a shift to meso- and macro-porosity as the removal of the exterior of the particle is sig-nificant at high burn-offs [\[27\]. T](#page-7-0)his shows that $CO₂$ was effective in creating well-developed pores on the surfaces of the precursor, hence leading to the activated carbon with a large surface area and porous structure which had high adsorption capacity for phenol.

3.4. BET surface area

The physical characterization of the best quality PAC produced was analyzed with Autosorb 1 surface area analyzer by nitrogen adsorption at −77 K. Prior to the analysis, the sample was degassed at 200 \degree C for 3 h. The surface area was calculated by the Brunauer, Emmett, and Teller (BET) equation [\[28\]](#page-7-0) using the nitrogen adsorption data. Using computer monitoring system, the adsorbed nitrogen volumes and various equilibrium pressures, and the BET surface area of the sample were reported. The PC software records the signals automatically and the pore volume and Isotherm were also obtained. The specific BET surface area of the best quality PAC produced was found to be $345.1 \text{ m}^2/\text{g}$. The results obtained agreed with previous study [\[29\]](#page-7-0) which reported that the BET surface is higher than 350 m²/g after carbonization of lignin at 900 °C. Furthermore, for both precursors, carbonization forms only micropores and mesopores, while macropores are generated only during the activation step. This phenomenon has been reported in other studies and may be explained by the fact that the activation not only forms the pores but also widens the size of the existing pores [\[30,31\].](#page-7-0)

3.5. Adsorption test with the activated carbon obtained by the optimum conditions and commercial activated carbon

A comparative adsorption study on total phenol removal was conducted between powdered activated carbon (PAC) prepared from EFB under optimum conditions and a commercial charcoal powdered activated carbon. The adsorption capacity (mg/g) and % removal of phenol by PAC produced and PAC commercial are shown in Table 6. The results showed that the prepared PAC from EFB and the commercial PAC prepared from charcoal are almost comparable in the adsorption of phenol. High adsorption of phenol, 4.8 mg/g by prepared PAC and 4.86 mg/g by commercial PAC was recorded after three minutes contact time. After 15 min, the performances were 4.81 and 4.9 mg/g by prepared and commercial PAC respectively. The adsorption then increased slightly to 4.87 and 4.9 mg/g after one hour contact time by prepared and commercial PAC respectively. There was a slight increase in performance by 1.2% and 1.8% for 3 and 15 min of contact times respectively by the commercial PAC compared to the selected PAC prepared from EFB respectively.

4. Conclusions

The activation temperature of the PAC produced was found to have the most significant effect on the yield and adsorption of phenol compared to the activation time and $CO₂$ gas flow rate. While the preparation conditions of 900 ◦C activation temperature, 15 min activation time and 0.1 L/min $CO₂$ gas flow rate gave the best quality PAC with higher adsorption capacity and yield. The performance of EFB-based PAC was found to be competitive to the commercial grade PAC. Therefore, the prepared activated carbon from EFB would be promising in such way: the industrial oil palm solid waste would be utilized for the production of activated carbons for the treatment of wastewaters including palm oil mill effluent (POME) which is known to have very high concentrations of phenolic compounds.

Acknowledgment

The authors are grateful to the Malaysian Ministry of Science, Technology and Innovation (MOSTI) for funding the research under eScience grant No. 03-01-08-SF0043.

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