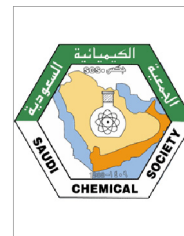




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# Optimization of preparation conditions for activated carbon from palm oil fronds using response surface methodology on removal of pesticides from aqueous solution

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## KEYWORDS

Palm oil fronds;  
Activated carbon;  
Optimization;  
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**Abstract** Palm oil fronds were used to prepare activated carbon using the physiochemical activation method, which consisted of potassium hydroxide (KOH) treatment and carbon dioxide ( $\text{CO}_2$ ) gasification. The effects of variable parameters activation temperature, activation time and chemical impregnation ratios (KOH: char by weight) on the preparation of the activated carbon and for the removal of pesticides: bentazon, carbofuran and 2,4-Dichlorophenoxyacetic acid (2,4-D) were investigated. Based on the central composite design (CCD), two factor interaction (2FI) and quadratic models were respectively employed to correlate the effect of variable parameters on the preparation of activated carbon used for the removal of pesticides with carbon yield. From the analysis of variance (ANOVA), the most influential factor on each experimental design response was identified. The optimum conditions for preparing the activated carbon from oil palm fronds were found as follows: activation temperature of 750 °C, activation time of 2 h and chemical impregnation ratio of 2.38. The percentage error between predicted and experimental results for the removal of bentazon, carbofuran and 2,4-D were 8.2, 1.3 and 9.2%, respectively and for the yield of the palm oil frond activated carbon was 5.6.

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## 1. Introduction

Pesticides are a group of artificially synthesized substances used to fight pests and improve the agricultural production. They are, however, generally toxic for living organisms and are difficult to degrade therefore, its toxic agents with persistent bio accumulative effects (El Bakouri et al., 2009). The use of pesticides also constitutes a risk for water quality in agricultural areas due to the fact that these components may

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pass through the soil and subsoil and pollute surface waters and groundwater.

Bentazon, carbofuran and 2,4-Dichlorophenoxyacetic acid (2,4-D) were widely used pesticides found in surface and ground water in the world. The World Health Organization (WHO) drinking water quality standard is 5, 3 and 20 µg/L for bentazon, carbofuran and 2,4-D, respectively. The wide range of pesticides in use makes research extremely difficult for producing a single method for the removal of herbicides or pesticides that applies universally (Gupta et al., 2006; WHO Guidelines for Drinking Water Quality, 2004).

There are several methods either independent or in conjunction that have been used for the removal of pesticides from water, such as chemical oxidation with ozone (Broséus et al., 2009), photocatalytic method (Lu et al., 2010), combined ozone and UV irradiation (Yeasmin et al., 2009), Fenton degradation (Yatmaz and Uzman, 2009), biological degradation (Chen et al., 2009), membrane filtration (Salman and Hameed, 2010), and adsorption (Midathana and Moholkar, 2009). However, the adsorption onto the activated carbon has been found to be superior compared to other techniques for wastewater treatment in terms of its capability for efficiently adsorbing a broad range of adsorbates and its simplicity of design. However, commercially available activated carbons are still considered expensive. This is due to the use of non-renewable and relatively expensive starting material such as coal, which is unjustified in pollution control applications (Salman et al., 2011).

A major challenge in the activated carbon production from new precursors is to produce very specific carbons which are suitable for certain applications. The most important characteristic of an activated carbon is its adsorption capacity which is highly influenced by the preparation conditions. Meanwhile, in assessing the effect of the preparation conditions on quality attributes, the use of an adequate experimental design is particularly important. Response surface methodology (RSM) has been found to be a useful tool to study the interactions of two or more variables (Karacan et al., 2007). Optimization of experimental conditions using RSM has been widely applied in various processes including the preparation of activated carbons (Baçaoui et al., 2001). Palm oil (*Elaeis guianensis*) grows well in wet, humid parts of the world, expectedly, large and abundant quantity of palm oil fronds are naturally generated by this process, which presently are underutilized and are often buried in rows within the palm plantations.

The objective of this research was to optimize the preparation conditions of activated carbon from palm oil fronds and its consequent application to remove bentazon, carbofuran and 2,4-D pesticides based on the RSM experimental design approach.

## 2. Experimental

### 2.1. Materials and equipment

Bentazon, carbofuran and 2,4-D (99.9%, 99.9% and 97.9% purity, respectively) obtained from Sigma-Aldrich were used as adsorbates. Distilled water was used to prepare all solutions. The surface morphology of the prepared activated carbon was examined using a scanning electron microscope (Model Leo Supra 50VP Field Emission, UK). The Brunauer-Emmett-Teller (BET) surface area, Langmuir surface area, total pore

volume and the pore size were measured using Micromeritics (Model ASAP 2020, US).

### 2.2. Preparation and characterization of activated carbon

Palm oil fronds are used as precursors for the preparation of activated carbon. The precursors were first cut into pieces (1–3 cm), washed with water to remove dirt from its surface and subsequently dried overnight at 100 °C. The dried precursors were crushed to the required size (1–5 mm) and then carbonized at 700 °C under purified nitrogen with a flow of 150 cm<sup>3</sup>/min for 2 h in a stainless steel vertical tubular reactor placed in a tubular furnace (the heating rate was fixed at 10 °C/min). The char produced was then soaked in potassium hydroxide (KOH) solution with different impregnation ratios (KOH: char). The mixture was then dehydrated in an oven overnight at 100 °C to remove moisture and then activated under carbon dioxide CO<sub>2</sub> atmosphere at different temperatures using stainless steel vertical tubular reactor placed in a tubular furnace (the heating rate was fixed at 10 °C/min). Once the final temperature was reached, the gas flow was switched over from nitrogen to CO<sub>2</sub> while activation was held for varying periods of time. The activated product was then cooled to room temperature and washed with hot distilled water, hydrochloric acid solution (0.1 M) and hot distilled water until the pH of the washed solution reached 6–7.

### 2.3. Adsorption studies

Batch adsorption was performed in 20 sets of 250 mL Erlenmeyer flasks. In a typical adsorption run, 100 mL of bentazon, carbofuran and 2,4-D solutions with initial concentration of 100 mg/L was placed in a flask. 0.30 g of the prepared activated carbon, with particle size of 2 mm, was added to the flask and kept in an isothermal shaker (120 rpm) at 30 °C until equilibrium was attained. The concentrations of pesticide solutions before and after adsorption were determined using a double beam UV-Vis spectrophotometer (UV-1700 Shimadzu, Japan). The maximum wavelength of the pesticides was found to be 333, 273 and 283 nm for bentazon, carbofuran and 2,4-D, respectively. The percentage removal of pesticides at equilibrium was calculated by the following equation:

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

where  $C_o$  and  $C_e$  (mg/L) are the concentrations of pesticides at initial and at equilibrium, respectively (Ayranci and Hoda, 2004).

### 2.4. Activated carbon yield

The experimental activated carbon yield was calculated based on the following equation:

$$\% \text{ Yield} = \frac{w_c}{w_o} \times 100 \quad (2)$$

where  $w_c$  and  $w_o$  are the dry weights of final activated carbon (g) and dry weight of precursor (g), respectively.

The prediction error resulting from the CCD in terms of prepared activated carbon yield and pesticide removal was evaluated as below:

$$\% \text{Prediction Error} = \frac{(\text{predicted} - \text{experimental})}{\text{predicted}} \times 100 \quad (3)$$

### 2.5. Design of experiments for preparation of activated carbon

The various process parameters for preparing the activated carbon were studied with a standard response surface methodology (RSM) design called a central composite design (CCD). This method is suitable for fitting a quadratic surface and it helps to optimize the effective parameters with a minimum number of experiments, and also to analyze the interaction between the parameters (Azargohar and Dalai, 2005). Generally, the CCD consists of  $2^n$  factorial runs with  $2n$  axial runs and  $n_c$  center runs (six replicates).

The activated carbons were prepared using the physiochemical activation method by varying the preparation variables using the CCD. The activated carbon preparation variables studied were (i)  $x_1$ , activation temperature; (ii)  $x_2$ , activation time and (iii)  $x_3$ , KOH: char impregnation ratio. These three variables together with their respective ranges were chosen based on the literature and preliminary studies. Activation temperature, activation time and chemical impregnation ratio are the important parameters affecting the characteristics of the activated carbons produced (Baçaoui et al., 2001). The number of experimental runs from the central composite design (CCD) for the three variables consists of eight factorial points, six axial points and six replicates at the center points indicating that altogether 20 experiments were required, as calculated from Eq. (4):

$$N = 2^n + 2n + n_c = 2^3 + 2 \times 3 + 6 = 20 \quad (4)$$

where  $N$  is the total number of experiments required and  $n$  is the number of process variables.

The experimental sequence was randomized in order to minimize the effects of the uncontrolled factors. The two responses ( $Y_1$ ) were activated carbon yield and pesticide removal ( $Y_i$ ). Each response was used to develop an empirical model which correlated the response to the three preparation process variables using a second degree polynomial equation (Zainuddin et al., 2005) as given by Eq. (5):

$$Y = b_o + \sum_{i=1}^n b_i x_i + \sum_{i=1}^n b_{ii} x_i^2 + \sum_{i=1}^{n-1} \sum_{j=i+1}^n b_{ij} x_i x_j \quad (5)$$

where  $Y$  is the predicted activated carbon yield or the removal response,  $b_o$  the constant coefficient,  $b_i$  the linear coefficient,  $b_{ij}$  the interaction coefficient,  $b_{ii}$  the quadratic coefficient and  $x_i$ ,  $x_j$  are the coded values of the activated carbon preparation or pesticide removal variables.

The activated carbon was derived from these precursors by the physiochemical activation method which involved the use of KOH treatment and followed by gasification with  $\text{CO}_2$ . The parameters involved in the preparation were varied using the response surface methodology (RSM). The three variables studied were:

- (1)  $x_1$ , activation temperature
- (2)  $x_2$ , activation time
- (3)  $x_3$ , KOH/char impregnation ratio (IR)

These three variables together with their respective ranges were chosen based on the literature and the results obtained

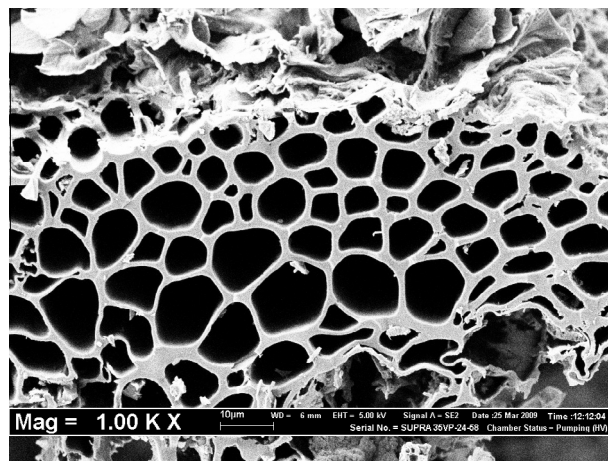
from the preliminary studies where the activation temperature, activation time and IR were found to be important parameters affecting the characteristics of the activated carbon produced (Karacan et al., 2007). The most important characteristic of an activated carbon is its adsorption uptake or its removal capacity which is highly influenced by the preparation conditions. Besides, the activated carbon yield during preparation is also a main concern in the activated carbon production for economic feasibility. Therefore, the responses considered in this study were:

- (1)  $Y_1$  activated carbon yield
- (2)  $Y_2$  removal of bentazon
- (3)  $Y_3$  removal of carbofuran
- (4)  $Y_4$  removal of 2,4-D

## 3. Results and discussion

### 3.1. SEM, BET and FTIR analysis

Fig. 1 shows the SEM image (magnification  $\times 1000$ ) of the activated carbon prepared under optimum conditions. It can be seen that the surface of the activated carbon prepared contains well-developed pores where there is a good possibility for pesticides (bentazon, carbofuran and 2,4-D) to be adsorbed into the surface of the pores. (BET) surface area, Langmuir surface area, total pore volume and the pore size were  $1237.13 \text{ m}^2/\text{g}$ ,  $1856.65 \text{ m}^2/\text{g}$ ,  $0.667 \text{ cm}^3/\text{g}$  and  $2.16 \text{ nm}$ , respectively. Fig. 2 illustrates the FTIR spectrum of the precursor and the activated carbon prepared. The surface chemistry of the activated carbon was different from the precursor as many of the functional groups disappeared after carbonization and activation processes. This was due to the thermal degradation effect during the carbonization and activation processes which resulted in the destruction of some intermolecular bondings. The main functional groups found on them were O–H stretching vibration of hydroxyl functional groups including hydrogen bonding which was detected at bandwidth around  $3405\text{--}3852 \text{ cm}^{-1}$ . Other major peaks detected are found at bandwidths of  $2929\text{--}2941$ ,  $1616\text{--}1644$ ,  $1400\text{--}1436$ ,  $1320\text{--}1321$ ,  $1010\text{--}1051$  and  $600\text{--}700 \text{ cm}^{-1}$  which were respectively assigned to C–H



**Figure 1** SEM image of palm oil fronds activated carbon prepared under optimum conditions (magnification =  $1000\times$ ).

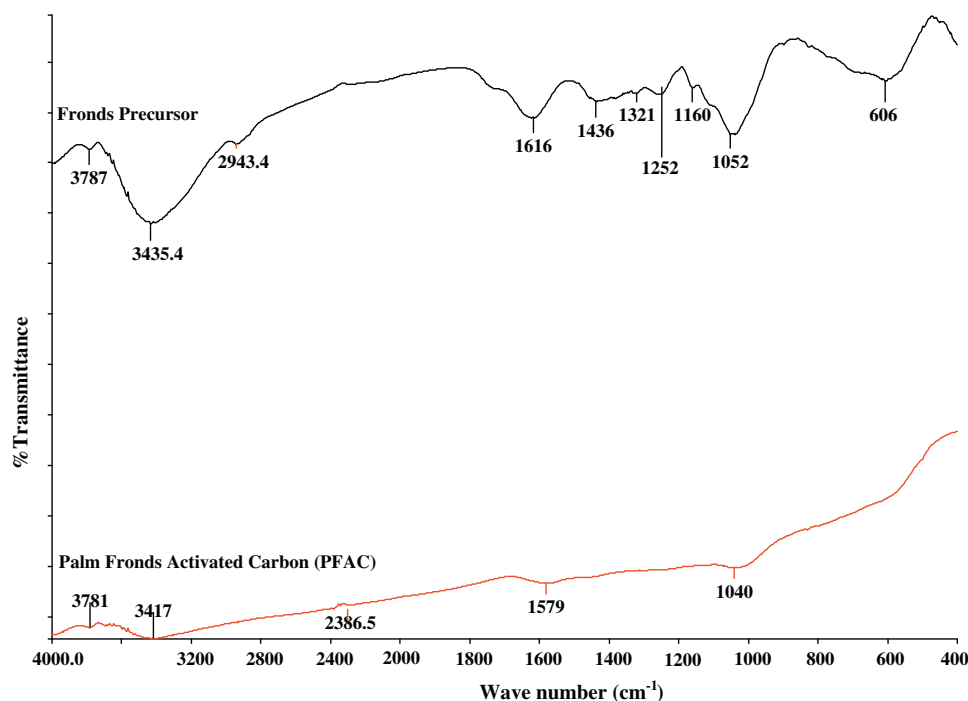


Figure 2 FTIR spectrums of fronds precursors and PFAC.

stretching related to alkanes and alkyl groups, C=C stretching in aromatics, CH<sub>2</sub> deformation in alkyl groups, Si-C stretching as well as C-O-C stretching vibrations in esters, ether or phenol groups. The medium peaks at 2360–2945 cm<sup>-1</sup> found on the spectrums of precursors, were assigned to the alkene group.

### 3.2. Preparation of palm oil frond activated carbon using DOE

The complete design matrix for the yield response of the activated carbon prepared from palm oil fronds with the four response values, carbon yield and pesticides (bentazon, carbofuran and 2,4-D) removal from the experimental works are presented in Table 1. Runs 15–20 at the center point were conducted to determine the experimental error and the reproducibility of the data.

The final empirical models in terms of coded factors (parameters) after excluding the insignificant terms for the activated carbon yield ( $Y_1$ ), bentazon removal ( $Y_2$ ), carbofuran removal ( $Y_3$ ) and 2,4-D removal ( $Y_4$ ) are given in Eqs. (6)–(9), respectively.

$$Y_1 = 17.90 - 4.79x_1 - 1.96x_2 - 1.865x_3 - 2.18x_1^2 - 0.66x_2^2 + 0.72x_3^2 - 1.56x_1x_2 + 0.36x_1x_3 + 1.16x_2x_3 \quad (6)$$

$$Y_2 = 93.44 + 4.45x_1 + 3.34x_2 + 9.49x_3 - 1.05x_1^2 - 0.36x_2^2 - 7.20x_3^2 - 4.62x_1x_2 - 4.00x_1x_3 - 3.70x_2x_3 \quad (7)$$

$$Y_3 = 94.27 + 4.29x_1 + 1.93x_2 + 8.03x_3 - 1.77x_1^2 - 0.055x_2^2 - 5.76x_3^2 - 2.75x_1x_2 - 3.50x_1x_3 - 3.72x_2x_3 \quad (8)$$

$$Y_4 = 93.8 + 4.51x_1 + 1.14x_2 + 5.51x_3 - 0.83x_1^2 - 0.32x_2^2 - 2.97x_3^2 - 2.06x_1x_2 - 4.69x_1x_3 - 1.56x_2x_3 \quad (9)$$

A positive sign in front of the terms indicates synergistic effect, whereas a negative sign indicates antagonistic effect. The quality of the model developed was evaluated based on the correlation coefficient value. The  $R^2$  values for Eqs. (6)–(9) were 96.25%, 88.49%, 88.97% and 90.70%, respectively. The closer the  $R^2$  value to unity, the better the model will be as this will give predicted values which are closer to the actual values for the response. The  $R^2$  of 0.9625 for Eq. (6) was considered relatively high, indicating that there was a good agreement between the experimental and the predicted in the carbon yield. The  $R^2$  of 0.885, 0.890 and 0.907 for Eqs. (7)–(9), respectively were considered high, indicating that there was an agreement between the experimental and the predicted values in the removal of bentazon, carbofuran and 2,4-D from this model. The adequacy of the models was further justified through analysis of variance (ANOVA). The ANOVA for the quadratic model for the activated carbon yield ( $Y_1$ ) is listed in Table 2.

The model  $F$ -value of 28.54 implied that this model was significant. Values of Prob >  $F$  of less than 0.05 indicate that the model terms were significant. In this case  $x_1$ ,  $x_2$ ,  $x_3$ ,  $x_1^2$ ,  $x_1x_2$  and  $x_2x_3$ , were significant model terms whereas  $x_2^2$ ,  $x_3^2$  and  $x_1x_3$  were insignificant to the carbon yield response. The ANOVA for response surface quadratic model for the bentazon removal ( $Y_2$ ) onto the prepared activated carbon is given in Table 3. The model ( $F$ -value) of 8.54 and (Prob >  $F$ ) of 0.0012 implied that this model was also significant. In this case,  $x_1$ ,  $x_3$  and  $x_3^2$  were significant model terms whereas  $x_2$ ,  $x_1^2$ ,  $x_2^2$ ,  $x_1x_2$ ,  $x_1x_3$  and  $x_2x_3$  were insignificant to the response. The ANOVA for the response surface quadratic model for the carbofuran removal ( $Y_3$ ) onto the prepared activated carbon is as listed in Table 4. The model ( $F$ -value) of 8.96 and (Prob >  $F$ ) of 0.0010 implied that this model was also significant. In this case,  $x_1$ ,  $x_3$  and  $x_3^2$  were significant model terms whereas  $x_2$ ,  $x_1^2$ ,  $x_2^2$ ,  $x_1x_2$ ,  $x_1x_3$  and  $x_2x_3$  were insignificant to the response. The ANOVA for the response surface quadratic model for

**Table 1** Preparation of palm oil frond activated carbon experimental design matrix and results for pesticides removal.

Run	Activated carbon preparation variables			% Activated carbon yield, $Y_1$	% Bentazon removal, $Y_2$	% Carbofuran removal, $Y$	% 2,4-D removal, $Y_4$
	Activation temperature, $x_1$ (°C)	Activation time, $x_2$ (h)	IR, $x_3$				
1	750	1.00	1.00	23.00	55.00	64.00	72.00
2	850	1.00	1.00	17.40	89.00	91.10	98.00
3	750	3.00	1.00	21.69	88.00	85.00	83.00
4	850	3.00	1.00	07.40	88.30	90.00	95.00
5	750	1.00	3.75	16.60	92.00	94.00	96.00
6	850	1.00	3.75	10.00	94.80	96.00	97.50
7	750	3.00	3.75	17.50	95.00	93.00	95.00
8	850	3.00	3.75	07.10	94.50	95.10	94.00
9	716	2.00	2.38	21.20	80.10	80.00	82.30
10	884	2.00	2.38	04.21	94.50	93.30	96.00
11	800	0.32	2.38	21.00	86.10	89.00	87.00
12	800	3.68	2.38	13.00	92.40	94.00	94.20
13	800	2.00	0.06	23.00	48.00	57.00	71.00
14	800	2.00	4.69	18.80	91.80	93.70	95.20
15	800	2.00	2.38	16.80	91.00	90.00	93.00
16	800	2.00	2.38	18.40	94.10	95.40	94.20
17	800	2.00	2.38	18.25	94.20	95.00	93.80
18	800	2.00	2.38	17.87	94.00	95.20	94.10
19	800	2.00	2.38	17.65	94.30	95.60	94.50
20	800	2.00	2.38	18.30	94.10	95.30	94.00

**Table 2** ANOVA for the activated carbon yield response (surface quadratic model).

Source	Sum of squares	Degree of freedom	Mean square	$F$ value	Prob > $F$
Model	530.03	9	58.89	28.54	< 0.0001
$X_1$	313.80	1	313.80	152.05	< 0.0001
$X_2$	52.45	1	52.45	25.42	0.0005
$X_3$	47.07	1	47.07	22.81	0.0008
$X_1^2$	68.54	1	68.54	33.21	0.0002
$X_2^2$	6.32	1	6.32	3.06	0.1106
$X_3^2$	7.40	1	7.40	3.59	0.0876
$X_1X_2$	19.50	1	19.50	9.45	0.0118
$X_1X_3$	1.04	1	1.04	0.51	0.4932
$X_2X_3$	10.83	1	10.83	5.25	0.0449

**Table 3** ANOVA results for bentazon removal onto prepared activated carbon.

Source	Sum of squares	Degree of freedom	Mean square	$F$ value	Prob > $F$
Model	2813.45	9	312.61	8.54	0.0012
$X_1$	270.84	1	270.84	7.40	0.0215
$X_2$	152.23	1	152.23	4.16	0.0687
$X_3$	1231.06	1	1231.06	33.65	0.0002
$X_1^2$	15.80	1	15.80	0.43	0.5259
$X_2^2$	1.84	1	1.84	0.05	0.8269
$X_3^2$	746.88	1	746.88	20.41	0.0011
$X_1X_2$	171.13	1	171.13	4.68	0.0559
$X_1X_3$	128.00	1	128.00	3.50	0.0909
$X_2X_3$	109.52	1	109.52	2.99	0.1143

the 2,4-D removal ( $Y_4$ ) onto the prepared activated carbon is also given in Table 5. The model ( $F$ -value) of 10.81 and (Prob >  $F$ ) of 0.0005 implied that this model was also

significant. In this case,  $x_1$ ,  $x_3$ ,  $x_3^2$  and  $x_1x_3$  were significant model terms whereas  $x_2$ ,  $x_1^2$ ,  $x_2^2$ ,  $x_1x_2$  and  $x_2x_3$  were insignificant to the response.



**Table 4** ANOVA results for carbofuran removal onto prepared activated carbon.

Source	Sum of squares	Degree of freedom	Mean square	F value	Prob > F
Model	1907.20	9	211.19	8.96	0.0010
$X_1$	251.17	1	251.17	10.62	0.0086
$X_2$	51.07	1	51.07	2.16	0.1725
$X_3$	881.53	1	881.53	37.27	0.0001
$X_1^2$	45.13	1	45.13	1.91	0.1972
$X_2^2$	0.043	1	0.043	0.01	0.9667
$X_3^2$	478.92	1	478.92	20.25	0.0011
$X_1X_2$	60.50	1	60.50	2.56	0.1408
$X_1X_3$	98.00	1	98.00	4.14	0.0692
$X_2X_3$	59.40	1	59.40	2.51	0.1441

### 3.3. Palm oil frond activated carbon yield

Experimental investigations revealed that the activation temperature has the greatest effect on the activated carbon yield. This was indicated by the response showing the highest *F* value of 152.05 as would be observed from Table 2. In the case of activation time and IR, similar effects were observed in the response except that they are less significant compared to activation temperature.

The quadratic effect of activation temperature on the yield of the prepared activated carbon was also higher compared to the quadratic effects of activation time and IR on the same response. Fig. 3(a) and (b) show the three-dimensional response and the interaction effects between parameters such as activation temperature, activation time and IR on the yield of the prepared activated carbon. Fig. 3(a) depicts the effect of activation temperature and activation time on the prepared activated carbon yield response with IR being fixed at zero level ( $IR = 2.38$ ) whereas Fig. 3(b) depicts the effect of activation temperature and IR on the same response where activation time was fixed at zero level ( $t = 2$  h).

In general, the yield of the prepared carbon was found to decrease with increasing activation temperature, activation time and IR. An increase in temperature would increase the release of volatiles as a result of intensification in dehydration and elimination reaction which would also increase the C–KOH and C–CO<sub>2</sub> reaction rate, thereby resulting in decreasing carbon yield (Adinata et al., 2007). An increase in the activation time also would cause more volatile matters to be released which lead to a decrease in carbon yield. Since KOH would promote the oxidation process, it therefore means that with

higher IR, the gasification of surface carbon atoms would be the predominant reaction, leading to an increase in the weight loss of carbon (Sudaryanto et al., 2006).

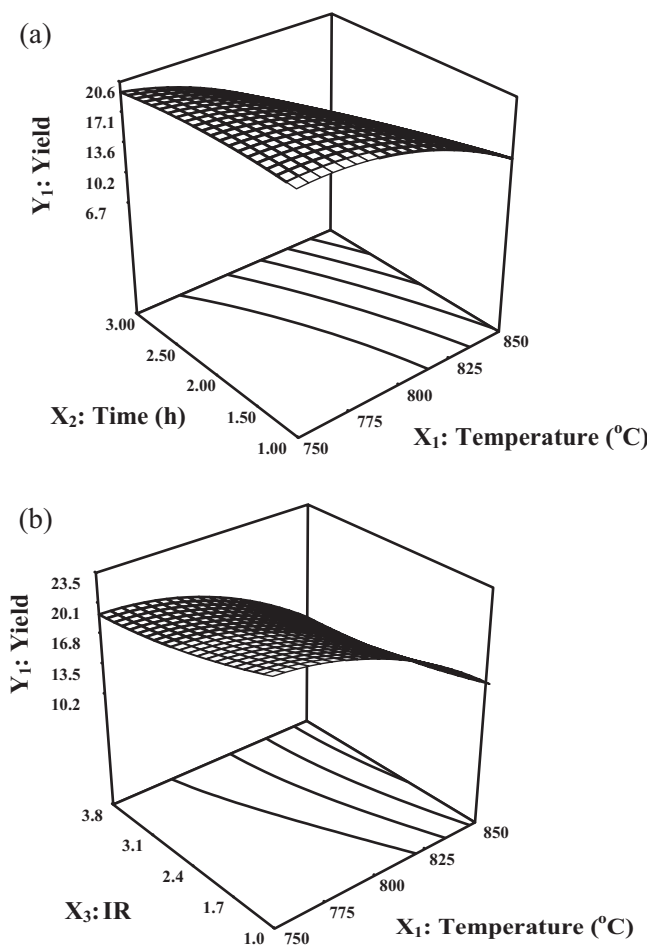
### 3.4. Bentazon, carbofuran and 2,4-D removal onto prepared activated carbon

The experimental observation for both the IR and activation temperature revealed that they have significant effects on the response of bentazon, carbofuran and 2,4-D removal onto the prepared activated carbon, whereas the activation time showed the least effect on this response. The quadratic effect of IR was also higher compared to the quadratic effects of activation temperature and time on the same response. The interaction effect between  $x_1$ ,  $x_2$  and  $x_3$  on the response of bentazon, carbofuran and 2,4-D removals were found to be moderate. Fig. 4(a–c) show the three-dimensional response and the interaction effects between the variable activation temperature, activation time and IR on the bentazon, carbofuran and 2,4-D removal.

These figures depict the effect of activation temperature and IR on the response surface with activation time being fixed at the zero level ( $t = 2$  h). It was observed from these figures that the removal of these pesticides on the prepared activated carbon generally increased with increasing activation temperature and IR. This is due to the formation of micropores caused by an increase in the activation temperature and also the development of porosity of the activated carbons prepared by KOH activation which is associated with gasification reaction. Similar results have been obtained by other researchers (Lua et al., 2006; Salman and Hameed, 2010).

**Table 5** ANOVA results for 2,4-D removal onto prepared activated carbon.

Source	Sum of squares	Degree of freedom	Mean square	F value	Prob > F
Model	1069.82	9	118.87	10.81	0.0005
$X_1$	277.31	1	277.31	25.23	0.0005
$X_2$	17.84	1	17.84	1.62	0.2315
$X_3$	414.07	1	414.07	37.67	0.0001
$X_1^2$	9.94	1	9.94	0.90	0.3641
$X_2^2$	1.45	1	1.45	0.13	0.7236
$X_3^2$	127.07	1	127.07	11.56	0.0068
$X_1X_2$	34.03	1	34.03	3.10	0.1090
$X_1X_3$	175.78	1	175.78	15.99	0.0025
$X_2X_3$	19.53	1	19.53	1.78	0.2121

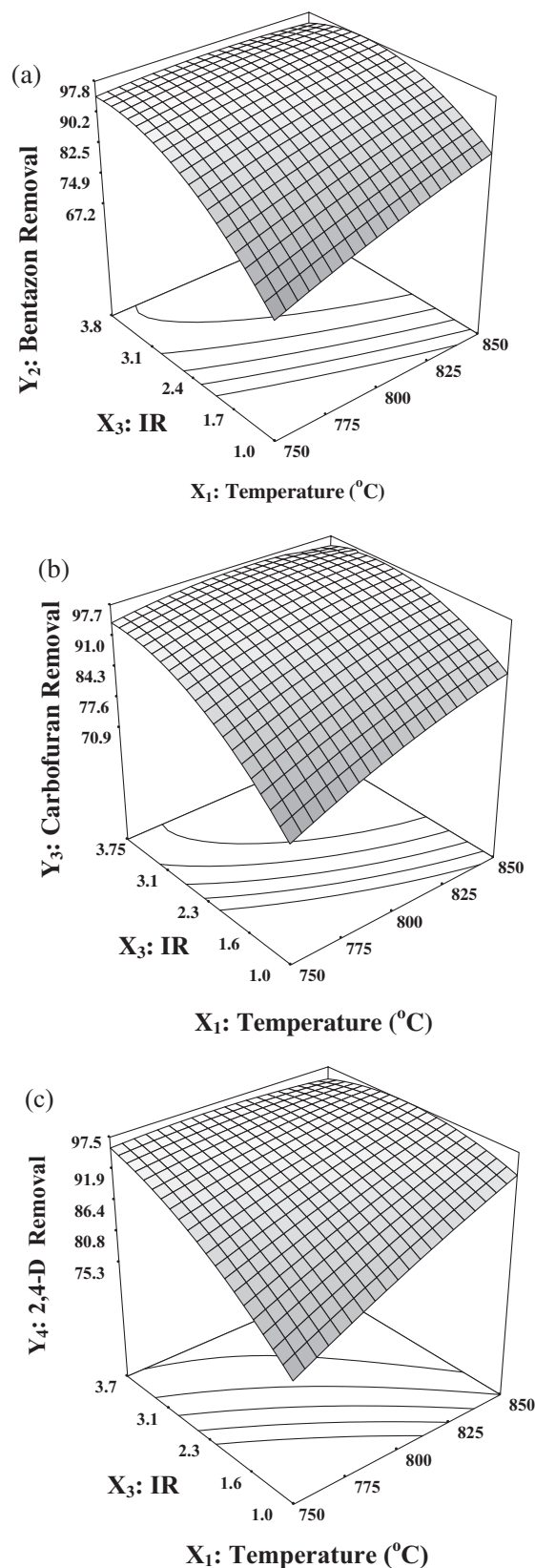


**Figure 3** Three-dimensional response on the yield of palm oil fronds activated carbon, the variable activation temperature and time (IR = 2.38), (b) the variable activation temperature and IR (activation time = 2 h).

### 3.5. Process optimization

The activated carbon produced should have accepted carbon yield and also a high pesticide removal for economical viability. However, to optimize these two response factors under the same conditions is difficult because the interest regions of the two factors were different. This phenomenon is expected, because the higher the KOH fraction used in the activation process, the higher will be the etching of the impurities and salts within the char matrix leaving behind a more porous but lighter activated carbon, thus recording higher adsorption capacities at the expense of activated carbon gross weight. Therefore, when  $Y_1$  increases,  $Y_i$  (for bentazon, carbofuran and 2,4-D) will decrease and vice versa. Therefore, in order to compromise between these two responses, the function of desirability was applied using Design Expert software version 6.0.6 (STAT-EASE Inc., Minneapolis, USA). The experimental conditions with the highest desirability were selected to be verified.

The predicted and experimental results of carbon yield and pesticide removal obtained at optimum condition are listed in Table 6. It can be found that the errors between predicted and



**Figure 4** Three-dimensional response between the variable activation temperature, time and IR for the removal of (a) bentazon, (b) carbofuran and (c) 2,4-D onto prepared activated carbon.

**Table 6** The optimum conditions for preparation palm oil fronds activated carbon and the percentage yield error of the predicted and experimental results for prepared activated carbon.

Activated carbon optimum preparation conditions			Activated carbon yield %		
Activation temp (°C)	Activation time (h)	IR	Predicted	Experimental	Error
750.0	3.00	3.75	19.1	20.5	5.6

experimental data for carbon yield, bentazon, carbofuran and 2,4-D removal are 5.6, 8.2, 1.3 and 9.2, respectively.

#### 4. Conclusions

Palm oil fronds were used as precursor to prepare an activated carbon with high surface area, sufficient yield of carbon and high pesticide removal. The optimum conditions for the preparation of the activated carbon using central composite design were found to be activation temperature of 750 °C, activation time of 2 h and chemical impregnation ratio of 2.38. Activation temperature was found to have the greatest effect on carbon yield.

The percentage errors between predicted and experimental results of carbon yield and pesticides removal at optimum condition were found to be 5.6, 8.2, 1.3 and 9.2, for carbon yield, bentazon, carbofuran and 2,4-D removal, respectively.

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