

Preparation of activated carbon from oil palm empty fruit bunch (EFB) by steam activation using response surface methodology

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Abstract: This paper aims to find the optimum conditions for preparation of activated carbon from empty fruit bunch (EFB) using physical steam activation by applying response surface methodology (RSM) with maximizing the BET surface area. The main process variables for this study were activation temperature and activation time and quadratic model was developed for a BET surface area which was selected as the investigated response. According to the RSM tool, the optimum conditions that were obtained from this study are at an activation temperature of 764.5°C and activation time of 77 min which give the maximum of BET surface area; 717.6 m²/g. Based on this optimum condition, the experimental value of BET surface area is 720.0 m²/g and is found to agree adequately with that (717.6 m²/g) predicted from the model.

Keywords: Oil Palm Empty Fruit Bunches, Steam Activation, Activated Carbon, Response Surface Methodology, BET Surface Area

1. Introduction

Activated carbon is widely used known as the most effective and useful adsorbents for the removal of pollutants from polluted gas and liquid because activated carbons have a large active surface area which can provide high adsorption capacity, well developed porous structures and good mechanical properties [1, 2]. Generally, activated carbons are produced by two-stage processes which are carbonization and followed by activation at high temperature either physically or chemically to produce an amorphous material with high surface area [3]. In physical activation process, the char that obtained from carbonization was activated with oxidizing gases such as steam or carbon dioxide at high temperature whereas for chemical activation process the starting materials are impregnated with a dehydrating agent [4].

Currently, researches interests are intensifying in the use of renewable source, low-cost and abundant lignocellulosic material as the common feedstock for the production of activated carbons [5]. In Malaysia, one of the promising

agricultural wastes to be utilized as an activated carbon is oil palm empty fruit bunch (EFB) because it is abundantly available in Malaysia and has very low high market value [6].

From previous researches, the activated carbons not only dependent on physio-chemical properties of starting materials but also dependent on the method preparation conditions which are suitable for certain applications [5]. In consideration of the treatment effects on characteristics of the manufacturing activated carbon, response surface methodology (RSM) has been applied as an experimental design in discovering the finest operating conditions. RSM is a very effective technique for this study because it presents statistical models which can be used to know the relationship between the parameters that have been optimized [2].

RSM was originally developed by Box and Wilson (1951) and has been found to be a useful tool to analyses the interactions of two or more variables [1]. RSM is useful for designing experiment, creating models, evaluating the effects of several variables and identifying the optimum conditions for desirable responses and helpful in reducing the number of experiments [7]. Though RSM is a popular

tool in process optimization, its application in EFB activated carbon production is has not been studied extensively for two operating variables. Some of the previous studies found, RSM is applied in preparation of activated carbons from precursors such as biochar, olive-waste cakes, Luscar char and Turkish lignite with three process variables [5,8].

Hence, the goal of this study is to utilize oil palm EFB for the production of activated carbon by steam activation and the application of RSM to identify the optimum preparation conditions for activated carbon with two operating variables, which gave the highest BET surface area. By considering the activation time and activation temperature as operating variables, a regression model for surface area (response) was developed.

2. Material and Methods

2.1. Raw Material

Empty fruit bunches (EFB) were collected from a palm oil producer (Meru Palm Oil Mill Sdn Bhd) in Klang, Selangor. The materials were cleaned with distilled water several times to remove dust and impurities.

2.2. Activated Carbon Preparation

The dried EFB (300g) was carbonized at 500°C for 1h in a turbular furnace, which consists of a vertical stainless steel reactor of length 35cm and internal diameter of 9cm in order to produce char. Then, the EFB char was activated by injection of steam at flow rate 120ml/hr at desired activation temperature and activation time. The experimental variables were activation temperature between the range of 750°C to 800°C and activation time of steam of range 60 min to 120 min. The activated carbon that was produced quantitative estimates for the BET surface area. AUTOSORB-1 (Quanta Chrome Instruments) was used in order to determine the BET surface area. The schematic diagram of experimental set-up for physical activation process is as shown in Fig. 1.

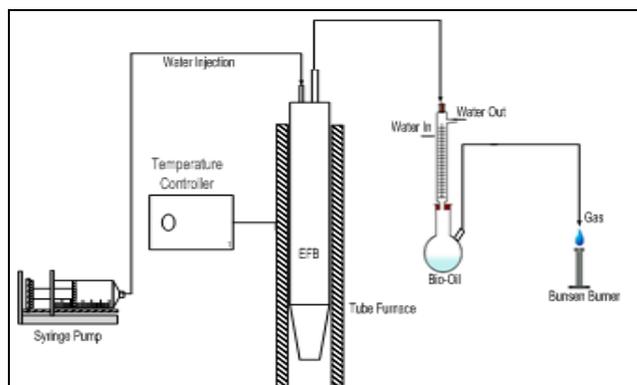


Figure 1. Experimental Set-up in Preparation of Activated Carbon

2.3. Response Surface Methodology

Central composite design (CCD) was applied to investigate a combine effect of several variables and to find

the optimum conditions for activated carbon production [7]. Activation temperature (A, °C) and activation time (B, min) were chosen as operating variables. BET surface area (Y, m²/g) was used as a response. The range and levels of the operating variables studied were selected are shown in Table 1. The value of α for this CCD was fixed at 1.414.

Table 1. High and low level of factors

Variables	Symbol	- α	-1	0	+1	+ α
Temperature (°C)	A	739.65	750	775	800	810.36
Time (min)	B	48	60	90	120	132

A full factorial CCD for the two variables, consisting of 5 factorial points, 4 axial points and 4 replicates at the center points are employed, with a total of 13 experiments were required, as calculated from the equation below.

$$N = 2^n + 2n + n_c = 2^2 + 2(2) + 5 = 13 \quad (2)$$

Where 'N' is the total of experiments, while 'n' is the number of independent variables.

The complete design matrix of the 13 experiments with the result obtained were performed in Table 2. The statistical software Design Expert 8.0.7.1 (STAT-EASE Inc., Minneapolis, USA) was used to analyse the data of the experimental design, analysis of variance (ANOVA) and regression analysis to fit the equations developed. The second order polynomial model was fitted for BET surface area obtained. Three-dimensional plots and their respective contour plots were obtained to study the interaction of one variable with another and the optimum conditions for preparation of activated carbon was identified.

Table 2. Experimental design matrix and results

Standard no.	Run no.	Temperature (°C)	Activation time (min)	BET surface area (g/m ²)
1	9	750	60	693.6
2	10	800	60	246.5
3	7	750	120	266.5
4	13	800	120	482.6
5	12	739.64	90	459.1
6	2	810.35	90	316.3
7	11	775	48	484.6
8	1	775	132	360.7
9	5	775	90	640.2
10	8	775	90	665.0
11	3	775	90	729.3
12	4	775	90	725.4
13	6	775	90	721.3

3. Result and Discussions

3.1. Development of Regression Model Equation

The value BET surface area that was obtained from experiments was found to range from 246.5 m²/g to 729.3 m²/g as shown as in Table 2. Based on the sequential model sum of squares, the highest order polynomials were used for

choosing the model, where the additional terms was significant and the model was not aliased [8]. As recommended by the software, quadratic model was selected

for BET surface area. Eq. (1) represents the regression model for response (BET) in terms of actual units.

Table 3. Analysis of variance (ANOVA) for selected model for BET surface area

Source	Sum of squares	DF	Mean Square	F Value	Prob > F	Status
Model	396937.514	5	79387.503	74.986	< 0.0001	Significant
A	23430.68	1	23430.68	22.132	0.0022	
B	16764.733	1	16764.733	15.835	0.0053	
AB	109958.56	1	109958.56	103.863	< 0.0001	
A ²	156498.262	1	156498.262	147.822	< 0.0001	
B ²	122155.784	1	122155.784	115.383	< 0.0001	
Residual	7410.835	7	1058.691			
Lack of Fit	723.143	3	241.048	0.144	0.9283	Not significant
Pure Error	6687.692	4	1671.923			
Cor Total	404348.349	12				
R ²	0.982					
Adj. R ²	0.969					
Pred. R ²	0.961					
Std dev.	32.54					

A: activation temperature (°C), B: activation time (min)

$$\text{BET} = 696.24 - 54.12 A - 45.78 B + 165.80 AB - 149.99 A^2 - 132.51 B^2 \quad (1)$$

where A is activation temperature (°C), B is activation time in minute (min) and BET surface area in m²/g, respectively. Positive sign in front of the terms AB indicates synergistic effect on BET surface area whereas negative signs of coefficients for A, B, A² and B² terms indicates antagonistic effects. The analysis of variance (ANOVA) is summarized in Table 3.

3.2. Statistical Model Analysis

The results show that the model predicted for the BET surface area was sufficient. The regression model obtained was significant ($P < 0.05$) with R² value of 0.982 and adjusted R² was 0.969. The closer the R² value to unity, the model will be of better quality and will give predicted values closer to the actual values for the response [9]. For predicted R², the value was 0.962 and is in reasonable agreement with the adjusted R². It demonstrates a satisfactory agreement between experimental and predicted values as the lack of fit F-value was insignificant for the model.

Analysis of variance (ANOVA) was further studied to justify the adequacy of model. The ANOVA is a sign whether the regression equation adequately represents the relationship between the response and significant variables [9]. The results of the ANOVA for the quadratic model of BET surface area activated carbon is also listed in Table 3. The Model F-value of 74.99 and Prob > F of <0.0001 implied that the developed model was significant. Values of "Prob > F" less than 0.05 indicated that the model terms were significant and if the values of "Prob > F" greater than 0.1 indicated the model terms were not significant. In this study, activation temperature (A), activation time (B), A², B² and AB were significant model terms to response, BET surface area.

Fig. 2 shows the predicted values against the experimental

values for BET surface area and from this figure it can be seen that these values were significantly close to each other. This indicates that the developed model was successful in capturing the correlation between the activated carbon preparation of chosen variables to BET surface area.

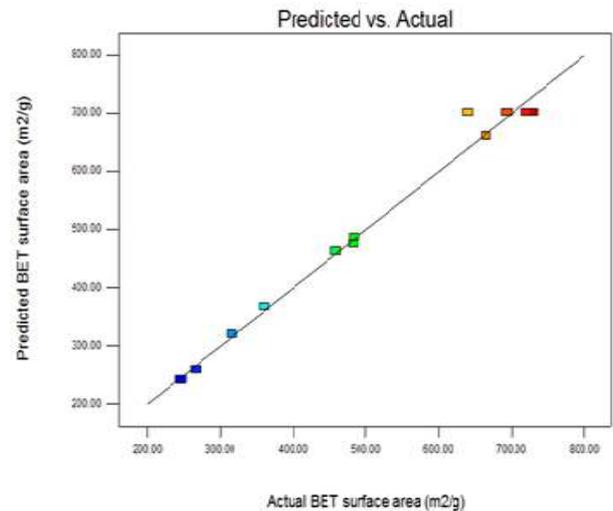


Figure 2. Predicted vs Experimental BET Surface Area.

3.3. Effect of Operating Variables on the BET Surface Area

Based on the ANOVA, the effects of two operating variables on the BET surface area of EFB activated carbon were investigated by using RSM three dimensional plots and contour plots. Fig. 3 and Fig. 4 show the response surface and contour plots for BET surface area with respect to physical activation temperature and activation time. Initially, the BET surface area increases with an increase in the activation temperature. This is because at high temperature, low-molecular weight volatiles were released

from the matrix structure and generated the fragile structure, resulting pore size increased [10]. However, as time breakdown occurred, BET surface area slowly decreased because the reaction was too extreme, so the micropores would have coalesced, resulting in mesopores [11]. According to Allwar, this may be caused by the excessive heat treatment and resulted the walls of porosities were broken and closed the mouth of pores [12]. The result obtained is agreement with research study reported by Alam *et al.*, (2008) that at high temperature, excessive heat transfer may cause shrinkage of activated carbon, resulting in narrowing or closing pores [13].

At long time activation, BET surface area was increased. This may be due at long time, the development of porosity was higher than a short time because steam was penetrated deeper inside of particles [11, 14]. However, when duration was too long, BET surface area was decreased. This may be due to the excessive carbon content burn-off which indirectly affects the quality of EFB activated carbon [1]. In addition, as the high activation temperature has to be maintained for a longer period of time, this will result on higher energy cost will be acquired.

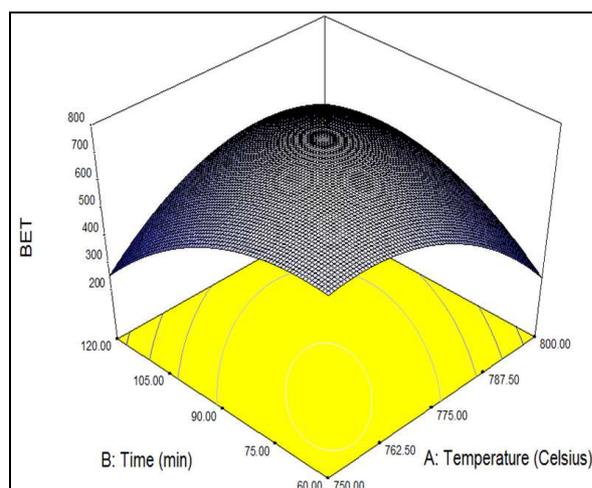


Figure 3. Response surface plot

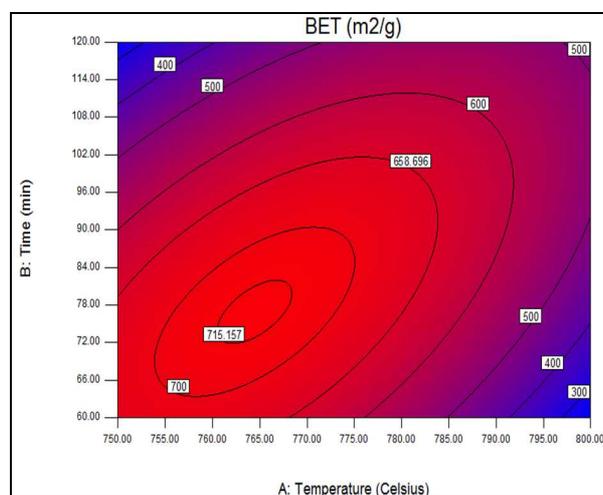


Figure 4. Contour plot showing the effects of activation temperature and activation time on BET surface area

3.4. Process Optimization

Optimization of the activation variables was identified using the optimizer function in the Design Expert software. The results are tabulated in Table 4. The optimum operating conditions calculated, physical steam activation temperature of 764.5°C and activation time of 77 min. Under the optimum activation conditions, BET surface area of EFB activated carbon that was predicted by the model and experimental are 717.6 m²/g and 720.0 m²/g, respectively. The optimum conditions of experimental and predicted results showed good agreement and reflects the existence of an optimal point.

Table 4. Optimum conditions with model validation

A (Temperature, °C)	B (Time, (min))	BET surface area (g/m ²)	
		Predicted	Experimental
764.46	77	717.6	720.0

4. Conclusion

Oil palm empty fruit bunch (EFB) can be used as the perfect precursor with steam as the activating agent for the preparation of activated carbon with high BET surface area. Statistical optimization of BET surface area on the activated carbon production has been successfully performed using RSM via DOE software by conducting a CCD by varying the activation temperature and activation time. The optimum conditions for preparation high BET surface area activated carbon were obtained as follows; activation temperature 764.5°C and activation time 77 min. Under these optimum conditions, the predicted value from the model of BET surface area is 717.6 m²/g and experimental values of BET surface area is 720.0 m²/g and this indicates the successful development model because small deviation error between of them.

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