

PREPARATION OF ACTIVATED CARBON PRODUCTION FROM OIL PALM EMPTY FRUIT BUNCH AND ITS APPLICATION

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ABSTRACT

Response surface methodology (RSM) with central composite design (CCD) was employed to optimize the activated carbon production from oil palm empty fruit bunch (OPEFB). First, carbonization was RSM-designed by carrying out in CO₂ with a focus on the effects of carbonization temperature and carbonization time. The maximum I₂ adsorption of 373.41 mg/g with surface area (BET) of 1.09 m²/g was presented by bio-char with carbonization condition of 450 °C for 90 min. Subsequently, the activation process was RSM-designed by H₃PO₄ activation. OPEFB bio-char was mixed with 85% (w/v) H₃PO₄ at bio-char/H₃PO₄ ratio (1:1-1:3 (w/v), activation temperature (400-600 °C) and activation time (60-180 min). The maximum I₂ adsorption of 841.32 mg/g and BET of 900.02 m²/g was presented by activated carbon in the activation condition of bio-char/H₃PO₄ ratio 1:2 at 668 °C for 120 min. In addition, the selected OPEFB-activated carbon was applied for furfural and 5-Hydroxymethyl furfural (5-HMF) adsorptions from the simulated solutions and it could remove of 139.94 and 18.79 mg/g, respectively.

KEYWORDS: activated carbon, oil palm empty fruit bunch, biomass, response surface methodology

1. Introduction

Activated carbons (ACs) are versatile adsorbent. One of the most important adsorbent commonly used in industries. Beside high surface area and micropore volume, favorable

pore size distribution make this group of adsorbent suitable for the removal of undesirable odor, color, taste, and organic and inorganic impurities from domestic and industrial waste water [1]. Activated carbon can be produced from nearly all carbon-containing organic materials, mainly lignocellulosic biomass; wood, sawdust, oil palm shell, oil palm empty fruit bunch, coal, lignite, etc [2-4]. Lignocellulose biomass (LB) is well known as biomass energy and become a valuable raw material for production of chemicals, polymers and sugars. LB is an efficient and economical material that means it is sustainable, renewable sources and cost-effective production [5]. Oil palm empty fruit bunches (OPEFBs) discarded from palm oil mills is a low cost substrate with highly abundant in several South-east Asian countries. Main characteristics of OPEFB contain about 30-60% cellulose, 15-35% hemicellulose and 4-20% and lignin on a dry weight basis [6-7].

In general, ethanol production using dilute acid hydrolysis of lignocellulose may result in sugars along with other byproducts from some serial and parallel reactions [8]. The performance of yeast, such as *Saccharomyces cerevisiae* and bacteria, such as *Zymomonas mobilis* [9-10], in lignocellulosic hydrolysates was correlated to the contents of 2-furfuraldehyde (furfural; from pentoses), 5-hydroxymethyl-2-furfuraldehyde (5-HMF; from hexoses) which subsequently degrade to furfural and 5-HMF [11]. The poor fermentability of dilute acid bagasse hydrolysates by *S. cerevisiae* relates to the high concentrations of fermentation inhibitors formed during severe acidic pretreatment [12]. There some materials used for recovery of furfural and 5-HMF such as Amberlite® polymeric resin XAD-4 [13], coconut-ACs [14] and zeolites [15].

The aim of this research is to optimize activated carbon production process from OPEFB by using statistical techniques for designing experiments (response surface methodology; RSM) in order to maximize furfural and 5-HMF adsorptions and highly BET surface area.

2. Materials and Methods

2.1 Materials

The OPEFB residue was obtained from Trang's palm oil extraction mill in Trang province, Thailand. The OPEFB were washed several times with running tap water and dehydrated overnight in an oven at 105°C and then OPEFB was ground to a particle size of

about 10-15 mm. Dried materials were kept inside plastic bags and kept in desiccators for 24 hours for removal of moisture and other volatile impurities.

2.2 Response surface methodology design

Response surface methodology (RSM) is generally used to investigate the combined effects of several variables and to find the optimum conditions for a multivariable system [16]. The central composite design (CCD) is one of the most commonly used response surface designs to study the effects of variables on their response, and subsequently in the optimization studies [17]. Optimization of OPEFB-biochar preparation was studied by using the Design expert software (Trial version 11.0, Stat-Ease, Inc., Minneapolis, USA) with CCD design matrix. The significance of each variable and their interactions, and fitting a predictive model to the experimental responses was based on the following second-order polynomial:

$$Y = \beta_0 + \sum_{i=1}^k \beta_i x_i + \sum_{i=1}^k \beta_{ii} x_i^2 + \sum_{i < j}^k \sum_j^k \beta_{ij} x_i x_j \quad (1)$$

Here Y is the observed response (xylose concentration, xylose yield, or digestibility of solid residue); β_0 is the constant term; i, j and k are integers (in this case i is from 1 to 3, j is from 2 to 3, and k is the total number of factors, 3); $\beta_i, \beta_{ii}, \beta_{ij}$ are, respectively, the coefficients for the linear, quadratic and interactive effects; and x_i and x_j are independent variables or factors, representing the acid concentration, hydrolysis temperature and hydrolysis time. The statistical software package Design Expert (Trial version 11.0) was used to analyze the results. The fit of the models was assessed from the coefficient of determination R^2 and the adjusted R^2 . Experimental validation of the model-based optimum set-point for acid hydrolysis was performed.

2.3 Optimization of OPEFB-biochar preparation using response surface methodology

Carbonization of OPEFB materials was conducted in a carbonization furnace. In detail, the furnace was first heated to the designated temperature and 100 g of OPEFB materials was then fed into the internal reaction zone of the furnace through the inlet tube at a constant rate by using N_2 of 1 L/min which was maintained throughout the carbonization and cooling stages to ensure O_2 -free conditions in the internal reaction zone of the furnace [18]. Two

independent variables, namely carbonization temperature (A, 400-500 °C) and carbonization time (B, 60-120 min) were used at five coded levels ($-\alpha$, -1, 0, +1, $+\alpha$), as shown in Table 1.

Table 1 Coded and real values of variables in the Central Composite design (CCD)

Independent variable	Unit	Code	Actual factor level				
			$-\alpha$	-1	0	+1	$+\alpha$
Temperature	(°C)	A	379	400	450	500	521
Time	min	B	48	60	90	120	132

2.4 Optimization of OPEFB-activated carbon preparation using response surface methodology

The OPEFB-biochar was chemical activation by using 30% ortho-phosphoric acid (H_3PO_4). Three independent variables were study of H_3PO_4 /biochar ratio (1:1-1:3 (w/v), activation temperature (400-600 °C) and activation time (60-180 min) and the five coded levels ($-\alpha$, -1, 0, +1, $+\alpha$), as shown in Table 2. In detail, 30 g of OPEFB-biochar powders were subsequently impregnated (H_3PO_4 /biochar ratio) 24 h of soaking duration. Next, the sample was filtered into the furnace and activated following the RSM-designed under nitrogen (N_2) atmosphere. After activation, the samples were allowed to cool down under N_2 flow before their remove from the furnace. The activated samples were washed in 3 M NaOH to remove the H_3PO_4 . Then, they were washed several times with hot distilled water, and finally with cold distilled water. The washed samples were dried at 105 °C for 24 h to prepare the activated carbons. Each sample was stored in sealed bottle and kept in desiccators.

Table 2 Coded and real values of variables in the Central Composite design (CCD)

Independent variable	Code	Actual factor level				
		$-\alpha$	-1	0	+1	$+\alpha$
Ratio	A	0.32	1	2	3	3.68
Temperature ($^{\circ}\text{C}$)	B	331	400	500	600	668
Time (min)	C	32	60	120	180	248

2.5 Batch adsorption test by using response surface methodology

The model inhibitors investigated in this work are furfural (99% purity, Sigma-Aldrich, St.Louis, MO), and 5-HMF (99% purity, Sigma-Aldrich, St. Louis, MO). Furfural and 5-HMF are highly toxic to ethanol fermentation microorganisms [19-20]. Concentrations of furfural and HMF were chosen so as to be close to those found in biomass hydrolyzate [21].

Batch absorption tests were performed to study the absorption behavior of the absorbents in the inhibitor-containing water solution. Furfural and HMF were added into the solution at concentration which was chosen so as to be close to those found in OPEFB hydrolyzate. Three independent variables, namely furfural and 5-HMF concentrations (3.0-6.0 g/L and 0.40-0.80 g/L, respectively.), absorption time (40-150 min) and pH (6.5-9.5) were used at five coded levels ($-\alpha$, -1, 0, +1, $+\alpha$), as shown in Table 3. The absorption batch tests were performed in the sealed glass vials at room temperature (30 ± 2 $^{\circ}\text{C}$). The glass vials were put in the shaker to agitate the samples. After mixing the carbon and liquid for the specified contact time, immediately filter each sample to separate the process liquid from the carbon. Analyze the filtrate to determine the inhibitor (furfural and 5-HMF) remaining in the solution.

Table 3 Coded and real values of variables in the Central Composite design (CCD)

Independent variable	Code	Actual factor level				
		$-\alpha$	-1	0	1	$+\alpha$
Furfural Conc. (g/l)	A	1.98	3	4.5	6	7.02
5-HMF Conc. (g/l)	A	0.26	0.4	0.6	0.8	0.94
Time (min)	B	2.5	40	95	150	187.5
pH	C	2.8	4.5	7.0	9.5	11.2

The furfural and 5-HMF concentrations in the aqueous solution were analyzed by using UV-spectrophotometer. A standard solution of the furfural and 5-HMF was scanned to determine the wavelength ($\lambda_{\max}$) corresponding to maximum absorbance which were 278 nm and 284 nm, respectively.

2.6 Sample characterization

The OPEFB-biochar and OPEF-activated carbon in each experiment were analyzed for density, yield according to AOAC methods [22]. While I_2 absorption, the iodine number is a technique employed to determine the absorption capacity of activated carbons. It indicates the porosity of the activated carbon and it is defined as the amount of iodine absorbed by 1 g of carbon at the mg level. The iodine number of activated carbon was obtained on the basis of the Standard Test Method, ASTM Designation D460786 by titration with sodium thiosulphate (ASTM, D 4607-86) [23]. The concentration of iodine solution was thus calculated from total volume of sodium thiosulphate used and volume dilution factor [24]. Morphology of OPEFB-biochar and -activated carbon was analyzed using a scanning electron microscope (SEM). The BET surface area (S_{BET}) was obtained by analyzing N_2 absorption according to the BET method [25].

3. Results and discussion

3.1 OPEFB-biochar preparation

The optimal OPEFB-biochar preparation parameters were experimentally determined using a CCD. The 2^3 factorial central composite experimental designs with six start points

and three replicates at the central point had 11 experimental runs in the design (Table 4). The observed value of OPEFB-biochar on density, yield and I_2 adsorption are shown with an average of the maximum experimental I_2 adsorption of 373.41 mg/g was obtained with carbonization temperature at 450°C for 90 min, while an average of the OPEFB-biochar density and an average of yield were 0.093 g/cm³ and 41.05%, respectively (Table 4) also, clearly presented in figure 1. The model F -value of 5.39 implies the model is significant. There is only a 4.41% chance that an F -value this large could occur due to noise. P -values of 0.0441 which less than 0.0500 indicate model terms are significant and the value of $R^2 = 0.8435$ indicates that 15.65 % of the total variation remains not explained by the model, so the correlation of experimental and fitted values is accepted (Table 5).

Table 4 Central composite design consisting of 11 experiments for the study of two experimental factors in coded units along with observed values

Run	Factor		Result		
	A: Temperature (°C)	B: Time (min)	Density (g/cm ³)	Yield (%)	I_2 adsorption (mg/g)
1	400	60	0.109	58.83	175.74
2	500	60	0.086	35.42	223.52
3	400	120	0.133	50.44	196.22
4	500	120	0.077	31.99	169.62
5	379	90	0.149	61.13	237.09
6	521	90	0.065	26.11	185.78
7	450	48	0.158	44.45	277.62
8	450	132	0.062	39.61	324.21
9	450	90	0.093	41.52	364.31
10	450	90	0.091	40.58	374.75
11	450	90	0.095	41.06	381.17

Table 5 Analysis of variance (ANOVA) for the fitted quadratic polynomial model

Source	F-value	P-value
Model	5.39	0.0441*
A : Temperature (°C)	0.1514	0.7132
B : Time (min)	0.0604	0.8156
AB	0.6344	0.4619
A ²	24.48	0.0043*
B ²	7.13	0.0443*

* Significant at $P < 0.05$

Even through the carbonization temperature (A) and carbonization time (B) were significantly effected but in this case, the twice time increasing of A² and B² are significantly decreased I₂ adsorption of OPEFB-biochar (Eq. 2).

$$I_2 \text{ adsorption (mg/g)} = 8429.489 - 0.039A^2 - 0.058B^2 \quad (2)$$

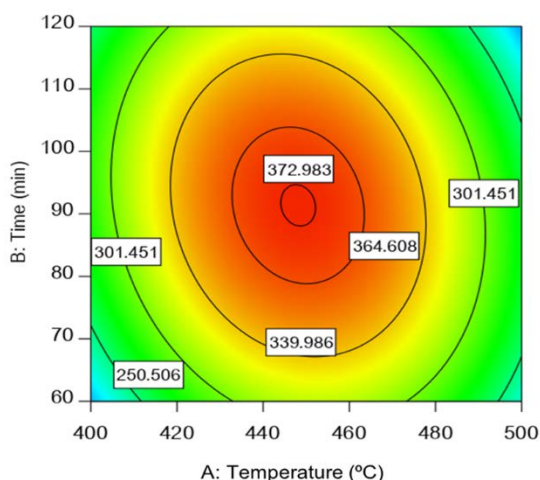


Figure 1 Contour plots of the interaction between carbonization temperature and carbonization time for OPEFB-biochar preparation on I₂ adsorption.

3.2 OPEFB-activated carbon preparation

The selected OPEFB-biochar produced from the optimal carbonization condition (Run 9-10, 3.1) was continually optimized for OPEFB-activated carbon preparation. The observed value of OPEFB-activated carbon on density, yield and I_2 absorption are shown in Table 6. The maximum experimental I_2 absorption of 841.32 mg/g was obtained with H_3PO_4 /biochar ratio of 1:2, activation temperature 668 °C and activation time of 90 min, while the OPEFB-biochar density and yield were 0.318 g/cm³ and 45.17%, respectively.

Table 6 Central composite design consisting of 17 experiments for the study of three experimental factors in coded units along with observed values

Run	Factor			Result		
	A: Ratio	B: Temperature (°C)	C: Time (min)	Density (g/cm ³)	Yield (%)	I_2 adsorption (mg/g)
1	1	400	60	0.328	72.25	170.9
2	3	400	60	0.322	67.81	332.72
3	1	600	60	0.333	54.32	265.6
4	3	600	60	0.305	51.02	549.1
5	1	400	180	0.324	65.27	224.75
6	3	400	180	0.333	60.64	587.08
7	1	600	180	0.329	38.44	200.05
8	3	600	180	0.317	30.26	680.8
9	0.32	500	120	0.354	63.92	76.06
10	3.68	500	120	0.337	59.27	316.5
11	2	331	120	0.327	60.15	302.9
12	2	668	120	0.318	45.17	841.32
13	2	500	32	0.303	60.72	507.02
14	2	500	248	0.31	58.07	381.2
15	2	500	120	0.331	61.23	481.2
16	2	500	120	0.34	62.92	471.5
17	2	500	120	0.357	60.25	474.3

The model F -value of 13.07 implies the model is significant. There is only a 0.13% chance that an F -value this large could occur due to noise. P -values less than 0.0500 indicate model terms are significant. The value of $R^2 = 0.9438$ indicates that 5.62 % of the total variation remains not explained by the model, so the correlation of experimental and fitted values is excellent (Table 7).

Table 7 Analysis of variance (ANOVA) for the fitted quadratic polynomial model

Source	F -value	P -value
Model	13.07	0.0013*
A: ratio	32.71	0.0007*
B: Temperature	35.31	0.0006*
C: Time	0.1107	0.7491
AB	2.31	0.1723
AC	3.73	0.0948
BC	1.29	0.294
A^2	28.01	0.0011*
B^2	3.99	0.0858
C^2	0.075	0.7922

* Significant at $P < 0.05$

In this study, H_3PO_4 /biochar ratio (A), activation temperature (B) and the twice time of H_3PO_4 /biochar ratio (A^2) were highly significant in their individual effects on I_2 absorption of OPEFB-activated carbon as shown in Eq. 3 and representative response surface plots are shown in figure 2A-2C.

$$I_2 \text{ adsorption (mg/g)} = 549.781 - 3.093A + 1.185B + 0.047A^2 \quad (3)$$

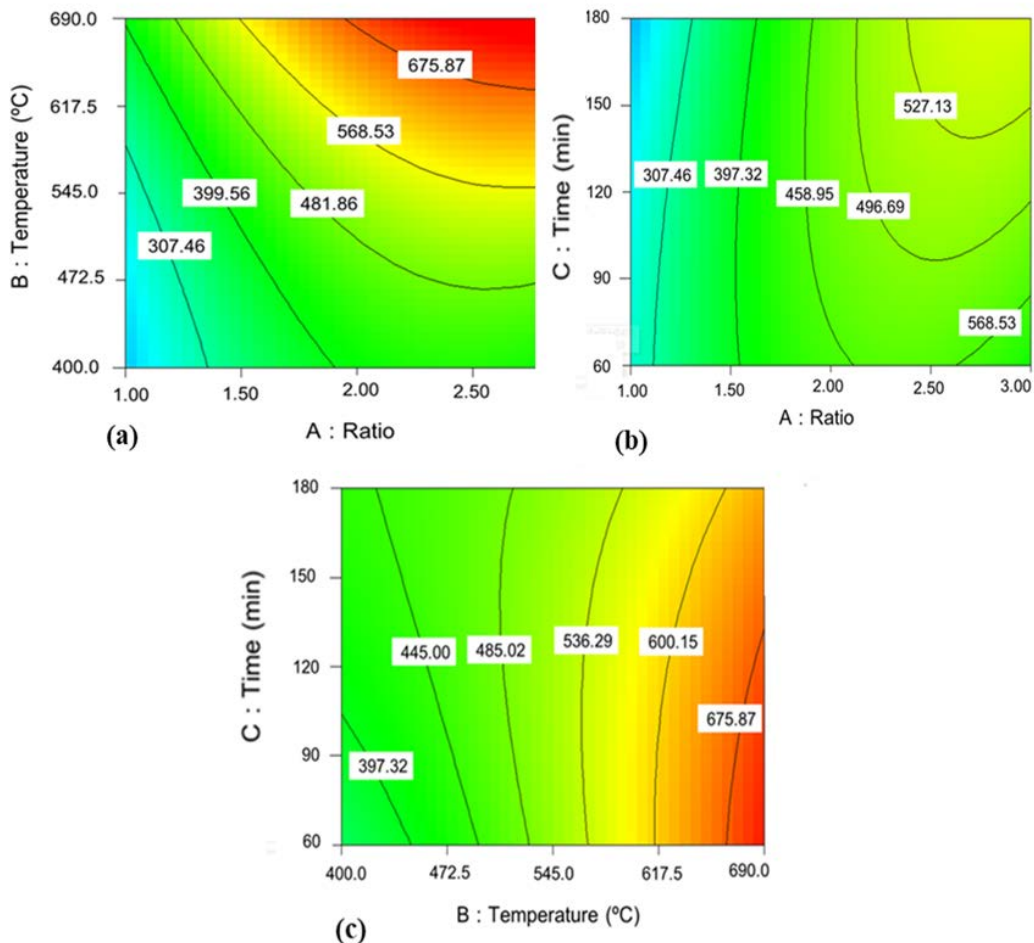


Figure 2 Contour plots for glucose production showing the interaction between (A) H_3PO_4 /biochar ratio and temperature; (B) H_3PO_4 /biochar ratio and activation time; and (C) carbonization temperature and carbonization time.

OPEFB-biochar and OPEFB-activated carbon yield showed that as temperature increase, the yield decrease. The higher yield at low temperature may be due to slow rate of carbon and carbon dioxide reaction with the weight loss resulted from the released of volatile matters [26]. The yield decreased quickly while the iodine number increased inversely, because most of volatile matter was diffused from granules in the first stage, so the steam penetrated easily into the surface of OPEFB; as a result, the high development of porosity [27]. From these results, the optimum temperature was 668°C since the iodine numbers of the resulted activated carbon were the highest of values. Moreover, our yield results also indicated higher

AC yield values than studies by Hameed et al. [28] (17-21% AC yield) while Alam et al. [29] found that activation temperature have the greatest effects to the activated carbon yield which opposite than our finding.

3.3 Removal of furfural and 5-Hydroxymethyl furfural

The separation of ethanol fermentation inhibitors (furfural and 5-HMF) from solutions (individually furfural and 5-HMF added solution) by OPEFB-activated carbon was optimized by using RSM. The effecting parameters; furfural or 5-HMF concentration, removal time and pH were optimized. The result showed that the maximal removal of both inhibitors was presented in the maximum furfural and 5-HMF concentrations (7.02 g/l and 0.94 g/l, respectively), removal time of 95 min and pH 7.0 (Table 8 and figure 3).

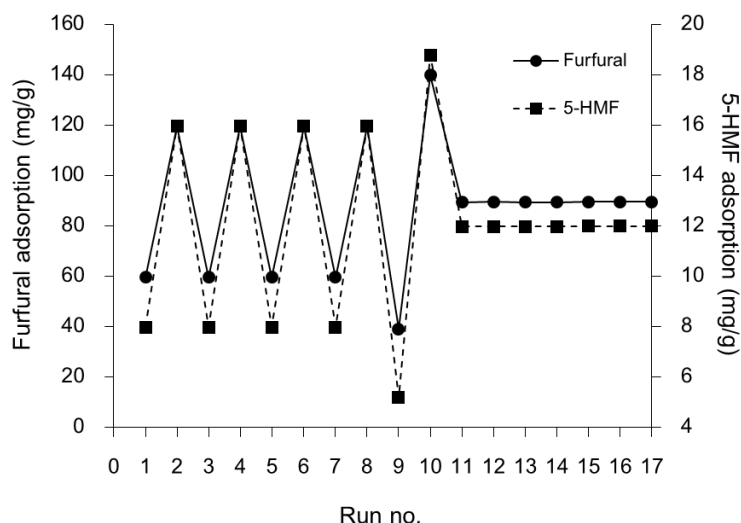


Figure 3 The effect of absorption conditions on the efficiency of furfural and 5-Hydroxymethyl furfural (5-HMF) absorptions for activated carbon from oil palm empty fruit bunch.

Table 8 Central composite design consisting of 17 experiments for the study of three experimental factors in coded units along with observed values

Run	Concentration		Time (min)	pH	Adsorption (mg/g)	
	(g/l)				Furfural	5-HMF
	Furfural	5-HMF				
1	3	0.4	40	4.5	59.58	7.92
2	6	0.8	40	4.5	119.55	15.98
3	3	0.4	150	4.5	59.58	7.99
4	6	0.8	150	4.5	119.54	15.97
5	3	0.4	40	9.5	59.57	7.98
6	6	0.8	40	9.5	119.54	15.98
7	3	0.4	150	9.5	59.57	7.98
8	6	0.8	150	9.5	119.54	15.98
9	1.98	0.26	95	7	39.19	5.18
10	7.02	0.94	95	7	139.94	18.79
11	4.5	0.6	2.5	7	89.54	11.98
12	4.5	0.6	187.5	7	89.56	11.98
13	4.5	0.6	95	2.8	89.54	11.99
14	4.5	0.6	95	11.2	89.02	11.97
15	4.5	0.6	95	7	87.56	11.89
16	4.5	0.6	95	7	89.55	11.96
17	4.5	0.6	95	7	89.55	11.99

Moreover, only inhibitors concentration was significantly effected on both furfural and 5-HMF removals (Table 9). In this case, the pH of the solution did not effect on absorption in both inhibitors. As mentioned by [30], acidic or basic solution is commonly used in the recovery of ionized chemicals which could be exchanged with H^+ or OH^- . However, furfural and 5-HMF are in the form of a non-ionized molecule in both solution and sorbent, thereby H^+ or OH^- is ineffective.

Table 9 shows the analysis of variance (ANOVA) for the fitted quadratic polynomial model for furfural and 5-HMF absorptions. Shortly described, in furfural absorption; the model F -value of 1181.80 implies the model is significant; there is only a 0.01% chance that a "Model F -Value" this large could occur due to noise. Values of "Prob > F " less than 0.0500 indicate model terms are significant. In this case is a significant model term. Values greater than 0.1000 indicate the model terms are not significant. The "Pred R^2 " of 0.9985 is in reasonable agreement with the "Adj R^2 " of 0.9985. While "Adeq_{Precision}" measures the signal to noise ratio which a ratio greater than 4 is desirable. This ratio of 122.341 indicates an adequate signal. Whereas, 5-HMF absorption; the Model F -value of 13316.26 implies the model is significant. The "Pred R^2 " of 0.9997 is in reasonable agreement with the "Adj R^2 " of 0.9999. "Adeq_{Precision}" measures the signal to noise ratio. This ratio of 410.828 indicates an adequate signal.

Table 9 Analysis of variance (ANOVA) for the fitted quadratic polynomial model

Source	Furfural adsorption		5-HMF adsorption	
	F -value	P -value	F -value	P -value
Model	1181.8	< 0.0001	13316.26	< 0.0001*
A: Furfural/5-HMF conc.	10627.77	< 0.0001	1.20E+05	< 0.0001*
B: Time	4.17E-05	0.995	0.14	0.7165
C: pH	0.052	0.8259	0.028	0.8727
AB	6.93E-06	0.998	0.43	0.5311
AC	6.93E-06	0.998	0.11	0.7515
BC	6.93E-06	0.998	0.24	0.6364
A^2	4.87	0.063	0.75	0.4163
B^2	4.8	0.0646	0.53	0.4916
C^2	3.58	0.1003	0.53	0.4916

* Significant at $P < 0.05$; $R^2_{\text{HMF}} = 0.9999$; Adj $R^2 = 0.9999$; Pred $R^2 = 0.9997$; Adeq_{Precision} = 410.828; $R^2_{\text{Furfural}} = 0.9993$; Adj $R^2 = 0.9985$; Pred $R^2 = 0.9985$; Adeq_{Precision} = 122.341

3.4 OPEFB-biochar and -activated carbon morphology

Scanning electron micrographs of the surface morphology of selected OPEFB-biochar and activated carbon samples are shown in figure 4(a)-(b). It can be seen from the micrographs that the external surface and inside holes of the OPEFB-biochar were smooth and without any cracks. On the other hand, the external surface and inside holes of the OPEFB-activated carbons were ruptured surface with a small number of pores and some grains in various sizes in holes and its surface. Furthermore, it contains an irregular and highly porous surface, indicating relatively high surface area. This observation can be supported by BET surface area of the OPEFB-biochar and -activated carbon samples as illustrated with 1.09 and 900.02 m²/g, respectively. In this results also indicated higher BET of our OPEFB-activated carbon than reported OPEFB-activated carbon by Hidayu et al. [3] which was prepared by physical steam activation. The BET surface area obtained was significantly high and it is within the acceptable range of commercial activated carbon (500-1500 m²/g). Generally, with high BET surface area, results in higher absorption capacity because the activated carbon is capable to absorb a number of gases under different conditions [31].

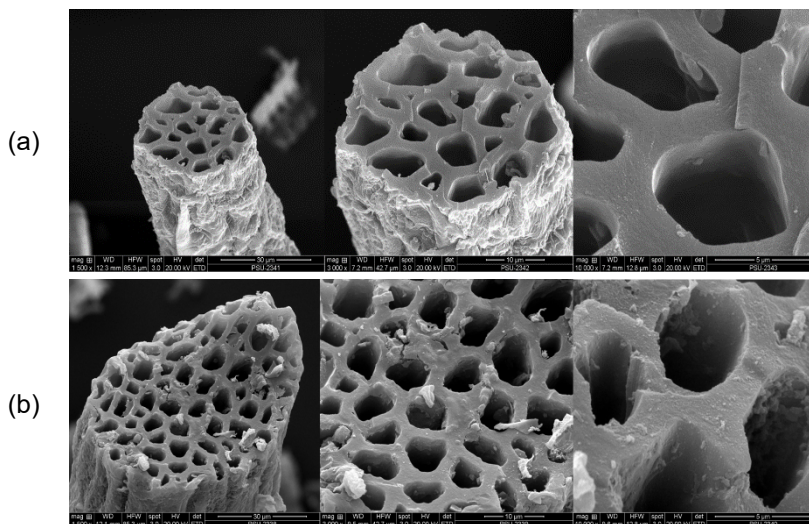


Figure 4 (a) Scanning electron micrographs of OPEFB-biochar preparation under optimum condition for carbonization temperature at 450°C for 90 min, (b) Scanning electron micrographs of OPEFB-activated carbon preparation under optimal condition by using H₃PO₄/biochar ratio of 1:2, activation temperature 668 °C and activation time of 90 min.

4. Conclusion

The optimization of activated carbon prepared under optimum conditions from oil palm empty fruit bunch (OPEFB) was analyzed in terms of I_2 adsorption and BET surface area. The I_2 absorption and BET surface area of selected OPEFB-activated carbon obtained was significantly high at 841.32 mg/g while the maximum separation of ethanol fermentation inhibitors (furfural and 5-HMF) from solutions (individually furfural and 5-HMF added solution) was 7.02 g/l and 0.94 g/l for furfural and 5-HMF, respectively. Moreover, the SEM micrographs of OPEFB-activated carbons presented of BET with 900.02 m²/g. This BET indicated that OPEFB was acceptable range of commercial activated carbon (500-1500 m²/g). As a conclusion, oil palm EFB can be utilized as a cheap raw material for the production of activated carbon.

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