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Optimization of activated carbon yield using the Taguchi method in synthesizing activated carbon from wood charcoal

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ABSTRACT

Wood charcoal was used as raw material for producing activated carbon. This research aims to determine the significant parameter and optimum preparation conditions resulting in the activated carbon yield. The activated carbon was prepared by the impregnation-activation method. In this process, potassium hydroxide has functioned as a chemical reagent. Three process parameters were varied such as KOH concentration (25-75 wt. %), activation temperature (600-850°C), and activation time (1-2.5 hours). The design of the experiment was arranged with the Taguchi method. For analysis, a signal-to-noise ratio was employed to obtain the optimal level, while the most significant process parameter was identified via ANOVA analysis. Based on the Taguchi orthogonal array design, the multiple regression model was developed to associate the process parameters with activated carbon yield. According to the results, activation temperature was the most significant parameter, while the optimum condition was 50 wt. % - 700°C - 2.5 hours. The predicted and experimental results were 87.3% and 67.6%, respectively. In conclusion, the results of this study designated that the Taguchi optimization method has efficacy in the synthesis of activated carbon.

ABSTRAK

Arang kayu telah digunakan sebagai bahan baku pembuatan karbon aktif. Tujuan penelitian ini adalah untuk mendapatkan parameter signifikan dan optimal dalam proses preparasi karbon aktif dengan *product yield* yang tinggi. Karbon aktif telah dibuat dengan metode *impregnation-activation* (IA). Dalam proses ini, kalium hidroksida telah difungsikan sebagai reagen kimia. Tiga parameter proses yang divariasikan yaitu konsentrasi KOH (25-75 wt. %), suhu aktivasi (600-850°C), dan waktu aktivasi (1-2.5 jam). Rancangan eksperimen didesain dengan metode Taguchi. Dalam analisis, *signal to noise* (S/N) *ratio* digunakan untuk mendapatkan level optimal, sedangkan parameter proses yang paling signifikan diidentifikasi dengan analisis ANOVA. Berdasarkan *Taguchi orthogonal array design*, *multiple regression model* dikembangkan untuk menunjukkan hubungan antara parameter proses dengan *activated carbon yield* (*AC yield*). Berdasarkan hasil tersebut, suhu aktivasi merupakan parameter yang paling signifikan untuk memaksimalkan *AC yield*, sedangkan kondisi optimum pembuatan karbon aktif adalah konsentrasi KOH 50 wt. %, suhu aktivasi 700°C, dan waktu aktivasi 2.5 jam. *Activated carbon yield* optimum berdasarkan prediksi dan hasil eksperimen adalah 87.3 dan 67.6%. Sebagai kesimpulan, hasil-hasil dari penelitian ini menunjukkan bahwa metode Taguchi memiliki efektivitas dalam proses sintesis karbon aktif.

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1. Introduction

In recent years, activated carbons (ACs) has been used in some applications, for instance, supporting catalyst [1], removing of pharmaceutical wastes [2], water treatment processing [3], water purification [4], supercapacitor [5], and energy storage material [6]. ACs were selected as valuable materials for these applications due to the lowest production cost, the highest carbon content, and the lower ash contents. These factors are essential evidence in producing activated carbons. Furthermore, materials-based wastes are used as natural raw materials such as wastes from biomass, wood, agriculture, and plantations [7].

Frequently, the activated carbon can be synthesized with three-step, i.e., dehydration, carbonization, and activation [8]. ACs can be activated in the activation step using diverse activation approaches, i.e., physical, chemical, and physicochemical. The essential difference between these approaches is the chemicals used and the process of compressing the gas. The physical approach comprises gas to activate chemical reagents such as steam, carbon dioxide, and air. While metal oxide, alkaline oxide, and acid are applied for the chemical approach. Some chemicals are the most widely used for chemical activation, such as zinc chloride ($ZnCl_2$), phosphoric acid (H_3PO_4), sodium hydroxide, and potassium hydroxide (KOH) [9].

Physical activation is processed in two steps: carbonization in the first and carbonization-activation in the second process. Chemical activation is a straightforward one-step procedure. The chemical reagent is imbued/mixed with carbon powder. As an activator, a chemical reagent is employed. After the impregnation process, the impregnated carbon powder is activated to form the porous structure of activated carbon materials. Chemical activation has the benefits of a lower temperature during carbonization and activation, a shorter carbonization period, and a formed porous structure than physical activation [10]. The chemical activation can be divided into two methods, i.e., carbonization-impregnation-activation (CIA) and impregnation-activation (IA) method. The most crucial property and characterization of activated carbon is its activated carbon yield (AC yield). The process parameters and preparation conditions influence the AC yield in the synthesis of activated carbon.

In this study, the activated carbons from wood charcoal were prepared with the impregnation-activation method using potassium hydroxide at different preparation parameters, i.e., concentrations of KOH (25, 50, and 75 wt. %), activation temperature (600, 700 and 850 °C), and activation time (1.0, 2.0 and 2.5 hours). The effects of these parameters on the yield of activated carbon were analyzed. The first objective of this work is to identify the best level for the KOH concentration, activation temperature, and the activation time to produce the activated carbon based on the Taguchi experimental design approach. The second purpose is to determine which factors significantly affect the yield of AC obtained from wood charcoal.

2. Material and Methods

2.1. Material

The material used to synthesize activated carbon was wood charcoal (WC) obtained from Spreading International Products Co., Ltd, Bangkok, Thailand. 85 wt. % potassium hydroxide and 0.1 N hydrochloric acid (HCL) purchased from PANREAC AppliChem were used for the chemical impregnation and activation process.

2.2. Preparation of the Activated Carbons

The wood charcoal bulk was crushed by conventional milling and sieved into varying particle sizes in the first step. The conventional milling was done by hand for crushing. Then the second step, the samples were milled into the smallest particle size using a planetary ball mill (SE-PM 4L). The applied milling parameters were 30 hours of milling time and two ball mill to powder ratio (BPR). The preparation of activated carbon was performed by the chemical-impregnation-activation method. The wood charcoal was impregnated with KOH using a 1 of impregnation ratio (IR). IR was the mass ratio between KOH and wood charcoal. The impregnation process was done on a hotplate magnetic stirrer (HMS-520). The mixed wood charcoal- KOH was then dehydrated in the drying oven (ECO CELL 22) at 120 °C for 18 hours. The dried samples were then activated in a muffle furnace (KL 0911/Ht40A). The activation process varied at different activation temperatures and times (Table 1). The activated carbons were cooled to room temperature, neutralized with a 0.1 M HCL solution, and washed with deionized water until the pH value reached 6 to 7. Finally, the activated carbons were dried at 120 °C for 24 hours. The preparation of the activated carbons from wood charcoal is shown in Figure 1.

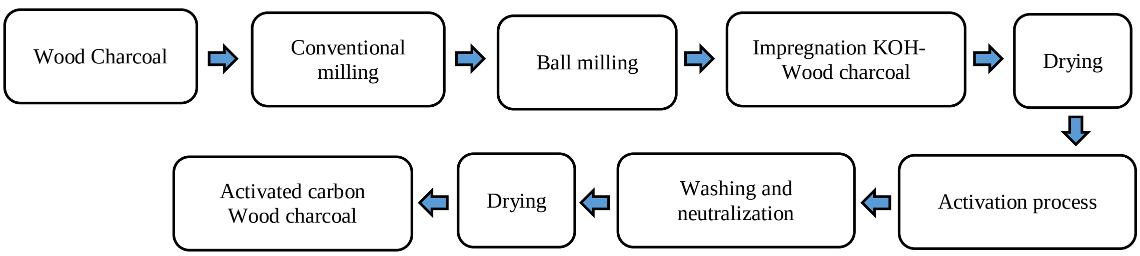

Figure 1. Flowchart for the preparation of activated carbon from wood charcoal.

2.3. Measurement of the Activated Carbon Yield

The experimental activated carbon yield was calculated based on the weight ratio between activated carbon and wood charcoal on a dry basis using an equation (1).

$$\text{Activated carbon yield (wt.\%)} = \frac{W_{ac}}{W_{wc}} . \quad (1)$$

where W_{ac} and W_{wc} are the dry weight of activated carbon (wt. %) and dry weight of wood charcoal (wt. %), respectively.

2.4. Experimental Design and Optimization

The preparation conditions for synthesizing the activated carbon were designed with the Taguchi method. It has been widely utilized in designing high-quality engineering systems. Some reasons for applying this method in the experiments are saving time, reducing costs, and discovering significant parameter processes quickly. Commonly, the Taguchi method has three basic steps, i.e., designing the system for determining the configuration, designing a parameter for determining the value of system parameters, and designing acceptance for determining the parameters' acceptance [11-15]. A statistical analysis using variance (ANOVA) analysis was employed to find the impact of process parameters on activated carbon yield. With the ANOVA analysis, the optimal process parameter levels in the synthesis of activated carbon with the highest yield of activated carbon can be estimated.

Taguchi method with an orthogonal array was selected with three activation process parameters at three levels. Table 1 shows process parameters (KOH concentration, activation temperature, and activation time) and their selected levels (25-75 wt.%, 600-850°C, 1.0-2.5 hrs). The output of this study was activated carbon yield. Three factors at three levels were investigated to observe the effect of process parameters on the activated carbon yield, as indicated in Table 2. In the synthesis of activated carbon produced from wood charcoal, a detailed flow diagram for the experimental design and analysis applied with the Taguchi method is shown in Figure 2.

Table 1. Activation process parameters in the synthesis of activated carbon.

Levels	**Process parameters**		
	KOH concentration, A (wt. %)	**Activation temperature, B (°C)**	**Activation time, C (h)**
1	25	600	1
2	50	700	2
3	75	850	2.5

Table 2. An orthogonal array of the Taguchi method for the activated carbon yield.

Run	**Designation**	**KOH concentration (wt. %)**	**Activation temperature (°C)**	**Activation time (h)**
1	$A_1B_1C_1$	25	600	1.0
2	$A_1B_2C_2$	25	700	2.0
3	$A_1B_3C_3$	25	850	2.5
4	$A_2B_1C_2$	50	600	2.0
5	$A_2B_2C_3$	50	700	2.5
6	$A_2B_3C_1$	50	850	1.0
7	$A_3B_1C_3$	75	600	2.5
8	$A_3B_2C_1$	75	700	1.0
9	$A_3B_3C_2$	75	850	2.0

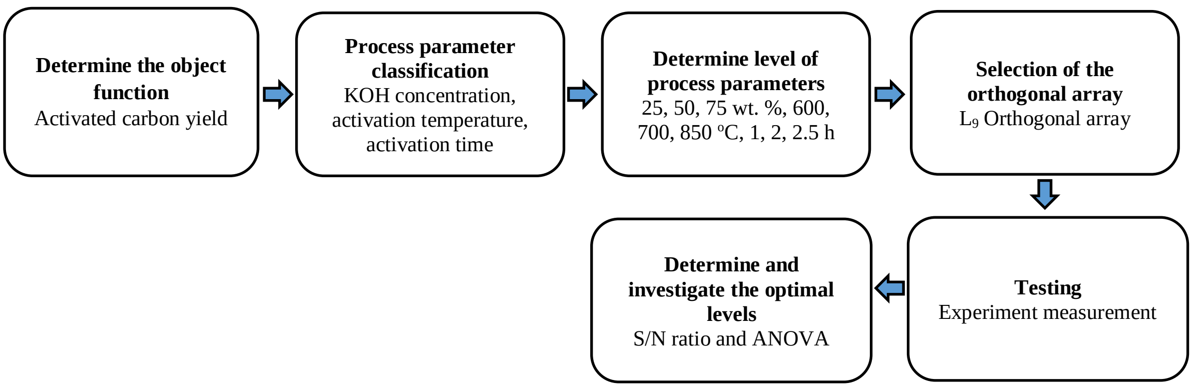

Figure 2. The Taguchi steps for the design of experiment and optimization.

3. Results and Discussion

3.1. Analysis of the Signal to Noise (S/N) Ratio

Table 3 shows the experimental results and the S/N ratio of activated carbon yield. The higher-the better response was selected to determine the optimum parameters condition is resulting in the highest activated carbon yield [10]. The optimal levels of processing parameters in the production of activated carbon yield were summarized in Table 4. The optimal levels were indicated by the highest value of response for S/N ratios of activated carbon yield. Figure 3 shows the graph for the S/N ratio response value. In Figure 3, the highest response value for activated carbon yield were 26.84 (KOH concentration), 33.37 (activation temperature), and 2.5 hours (activation time). According to the response for S/N ratios, it can be seen that the maximum activated carbon was obtained at a KOH concentration of 50 wt. %, activation temperature of 700 °C, and activation time of 2.5 hours. It can be concluded that the conditions of the process parameters for the optimal activated carbon yields were designed with the same condition at run no. 5, i.e., $A_2B_2C_3$.

Figures 4 show an interaction plot for each factor (KOH concentration, activation temperature, and activation time) in which a parallel line denotes no interaction (effect). In contrast, a crossing line indicates significant interaction. Figure 4 presents the interaction for activated carbon yield. KOH concentration of 25 wt. % interacts with 50 wt. % at 850 °C and 75 wt. % at 800 °C. Besides, KOH concentration of 25 wt. % interacts with 50 wt. % at 1.5 hours and 75 wt. % at 1.5 and 2.25 hours. The interaction between all KOH concentrations of 25, 50, and 75 wt. % was in 1.5 hours. In Figures 4, it can be seen that the interaction of KOH concentration shows the most significant influence on other parameters, followed by activation temperature, which has a significant influence on the activation time. An interaction plot with a more parallel line will have a significant effect on the process.

Table 3. The experimental results and S/N ratio values of activated carbon yield.

Run	Designation	KOH concentration (wt. %)	Activation temperature (°C)	Activation time (h)	Activated carbon yield (wt. %)	S/N ratio (dB)
1	$A_1B_1C_1$	25	600	1.0	27.2	28.6914
2	$A_1B_2C_2$	25	700	2.0	29.39	29.3640
3	$A_1B_3C_3$	25	850	2.5	12.25	21.7627
4	$A_2B_1C_2$	50	600	2.0	42.9	32.6491
5	$A_2B_2C_3$	50	700	2.5	67.6	36.5989
6	$A_2B_3C_1$	50	850	1.0	12.1	21.6557
7	$A_3B_1C_3$	75	600	2.5	42.5	32.5678
8	$A_3B_2C_1$	75	700	1.0	51	34.1514
9	$A_3B_3C_2$	75	850	2.0	4.9	13.8039

T_{PY} = Total mean value of activated carbon yield = 32.20%; $T_{PY\text{-}S/N}$ = S/N ratio total mean value = 27.9161 dB.

Table 4. Response for S/N ratios of activated carbon yield.

Levels	Process parameters		
	KOH concentration, A (wt. %)	**Activation temperature, B (°C)**	**Activation time, C (h)**
1	26.61	31.30	28.17
2	**30.30**[a]	**33.37**[b]	25.27
3	26.84	19.07	**30.31**[c]
Delta	3.70	14.30	5.04
Rank	3	1	2

a, b, c were the highest values of S/N ratios response

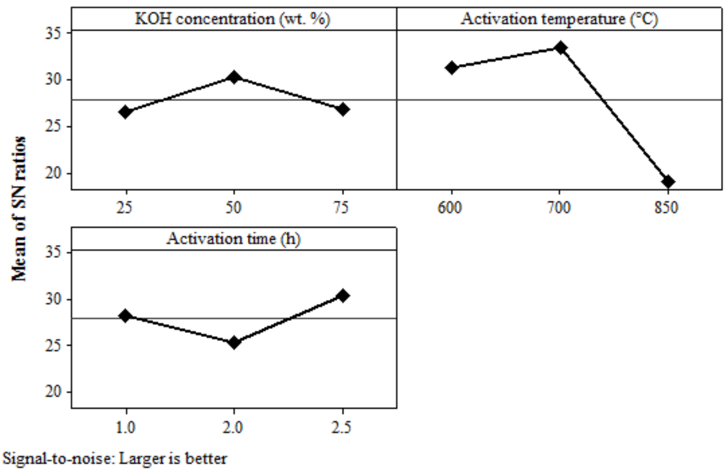

Figure 3. Effect of process parameter on S/N ratio for activated carbon yield.

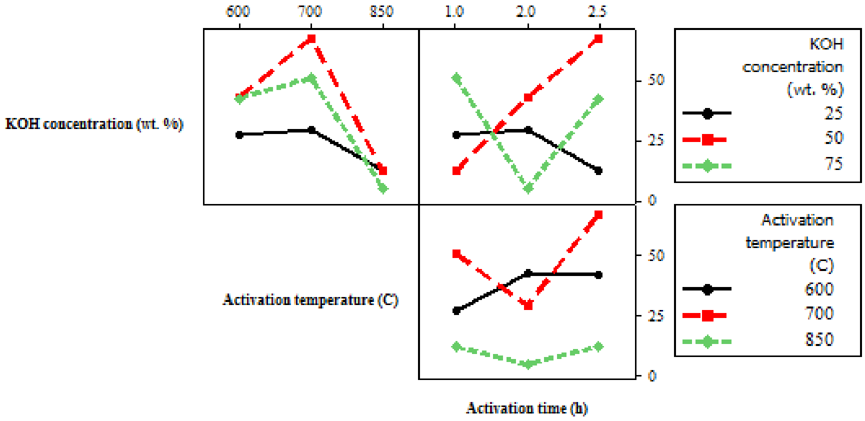

Figure 4. Interaction plot of process parameters in the synthesis of activated carbon.

3.2. Analysis of Experimental Results

Figure 5 shows the activated carbon yield resulted from the process parameters conditions. The yield of activated carbon increased from 25 to 50 wt. % of KOH concentration, 600 to 700 °C of activation temperature, and 1 to 2 hours of activation time. Then it decreased to 75 wt. %, 850 °C, and 2.5 hours. The highest activated carbon yield was reached at level 2 of all process parameters, i.e., 50 wt. % of KOH concentration, 700 °C of activation temperature, and 2 hours of activation time.

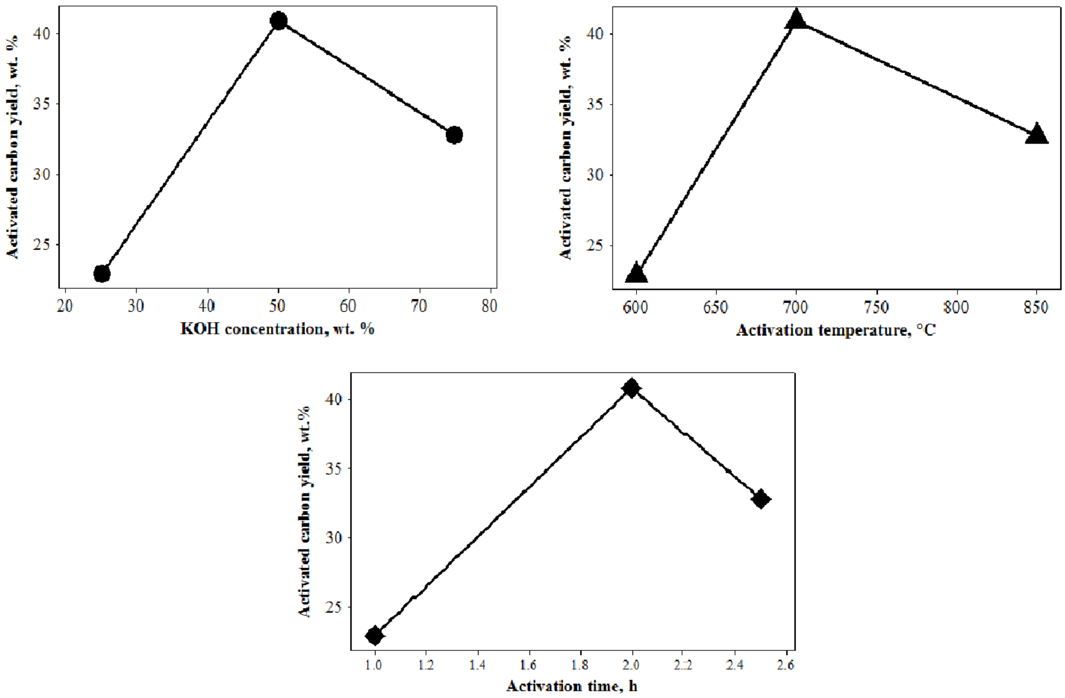

Figure 5. Effect of process parameters on the activated carbon yield.

3.3. Analysis of Variance (ANOVA)

ANOVA was applied to analyze the effects of process parameters such as KOH concentration, activation temperature, and activation time on activated carbon yield. The ANOVA results are shown in Table 5. According to the ANOVA results, the F-value of KOH concentration, activation temperature, and activation time were 5.58, 28.61, and 4.15, respectively. From these values, it can be concluded that the most important process parameter affecting the activated carbon yield was activation temperature (B) due to the highest F-value. The F-test principle indicated that the larger the F-value, the greater effect on the performance characteristic due to the change in that variable. Significance levels were also mentioned based on the P-value. Significance levels consist of suitable and unsuitable. The lowest P-value, the most significant level, and the effect of the process parameters. P-value confirmed that significance level activation temperature > KOH concentration > activation time i.e. 0.034, 0.152, and 0.194. The high of R-Sq, i.e., 97.46%, showed that the results are statistically acceptable.

Table 5. Analysis of variance results of activated carbon yield.

Source	DF	Seq SS	Adj SS	Adj MS	F	P	Significance level
A	2	483.29	483.29	241.64	5.58	0.152	Level 2
B	**2**	**2477.65**	**2477.65**	**1238.83**	**28.61**	**0.034**	**Level 1**
C	2	359.83	359.83	179.92	4.15	0.194	Level 3
Error	2	86.61	86.61	43.30			
Total	8	3407.38					

S=6.58, R-Sq = 97.46%, R-Sq (adj) = 89.83%

3.4. Regression Analysis of Activated Carbon Yield

The multiple regression model was applied in finding the predictive equation for activated carbon yield. According to the summary output of the multiple regression model in Table 6, the equation for the predicted values of activated carbon yield is shown in Equation (2).

Table 6. Summary of multiple regression model of activated carbon yield.

Predictor	Coef	SE Coef	T statistic	P value
Constant	100.59	49.50	2.03	0.098
KOH concentration	0.197	0.302	0.65	0.543
Activation temperature	-0.123	0.060	-2.05	0.096
Activation time	5.480	9.899	0.55	0.604

$$\text{Predicted activated carbon yield} = 101 + 0.197^{*}\mathrm{A} - 0.123^{*}\mathrm{B} + 5.58^{*}\mathrm{C}\ . \qquad (2)$$

where A, B, and C are KOH concentration, activation temperature, and activation time, respectively.

3.5. Estimation of Optimum Activated Carbon Yield

The optimization process in the Taguchi method was applied in getting the optimum activated carbon yield using Equation (3). The level for optimum activated carbon yield is found from the response of the S/N ratio in Table 4.

$$\text{Activated carbon yield} = \left(\left(\mathrm{A_{opt}} - \mathrm{T_{py}}\right) + \left(\mathrm{B_{opt}} - \mathrm{T_{py}}\right) + \left(\mathrm{C_{opt}} - \mathrm{T_{py}}\right) + \mathrm{T_{py}}\right)\ . \qquad (3)$$

where A_{opt}, B_{opt}, C_{opt}, and T_{py} are the optimal level from KOH concentration, activation temperature, activation time, and the total mean value of activated carbon yield, respectively. By applying this equation, it was calculated that the optimum activated carbon yield is 87.31%.

3.6. Confirmation Tests

In the Taguchi method, an experimental confirmation test is applied to verify the estimated results in the final step. The optimal condition is set for the significant factors. The results from the confirmation test are compared with the predicted based on the parameters and levels tested. In this study, a confirmation test was conducted by setting the optimal process parameters for activated carbon yield, i.e., A2B2C3 (Table 7).

Table 7. Results of the confirmation experiment for activated carbon yield.

	Optimal process parameters	
	Prediction	**Confirmation experiment**
Level	$A_2B_2C_3$	$A_2B_2C_3$
Activated carbon yield (wt. %)	87.3	67.6
S/N ratio (dB)	38.15	36.59

4. Conclusions

Taguchi method was designed to evaluate the effects of concentration of chemical reagent, activation temperature, and activation time to maximize the activated carbon yield. The optimization process was carried out, and the experimental results obtained were found with the predicted values. The maximum AC yield, i.e., 87.3 %, was found as 50 wt. %, 700 °C, and 2.5 hours. The interaction plot showed that the activation temperature affected the activated carbon yield more significantly compared with KOH concentration and activation time. As the plan, the applied method formulated from this study would be implemented in further research, especially in producing high product yield activated carbons.

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