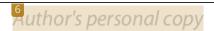
Taguchi method for optimizing process parameters in the production

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ORIGINAL ARTICLE



Taguchi method for optimizing process parameters in the production of activated carbon from rubber seed shell

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Abstract

Activated carbon is widely used in many applications. This study aims to optimize the most influential processing parameters of the activated carbon production from rubber seed shell. The Taguchi method is applied to gain 9 combinations of experimental design from three processing parameters, namely chemical reagent concentration, activation temperature, and activation time. By using the analysis of variance (ANOVA), the most influential processing parameters of activated carbon production are found as chemical reagent concentration and activation temperature. The optimal product yield and amorphous percentage can be achieved when chemical reagent concentration, activation temperature, and activation time are chosen as 25 wt.%—600 °C—1.0 h and 25 wt.%—600 °C—2.5 h, respectively. As conclusions, the product yield is influenced by the activation temperature, while the amorphous percentage is affected by the activation time. The Taguchi method is an appropriate approach for optimizing the parameters to produce the activated carbon.

Keywords Activated carbon · Amorphous percentage · Analysis of variance · Product yield · Taguchi method

1 Introduction

For many years, carbon-based materials (CBMs) and activated CBMs (ACBMs) have received considerable attention from scientists and researchers. Due to the essential characteristics, i.e., high surface area and a high degree of porosity, ACBMs are widely considered to be one of the most important materials in many fields, such as water treatment process, the removal of harmful gases [1], and engineering applications (super-capacitor, catalyst, energy storage). Some CBMs are obtained from natural resources such as agricultural byproduct [2] including almond shell, hazelnut shell, walnut shell [3], corncob [4], rice husk [5], potato peels [6], palm shells and *Jatropha curcas* fruit shells [7], cashew shells and millet stalks [8], and farming wastes [2, 9], which offer low-cost production and abundant amount.

In America, West Africa, the Indian sub-continent, and Southeast Asia, around the tropics, there are many rubber plantations. In the Southeast Asia region, Indonesia has the

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largest rubber plantation area in a total of 3.4 million hectares compared with 2.67 million hectares in Thailand and 1.02 million hectares in Malaysia. The rubber seed shells (RSS) are plenty but inedible in Indonesia; they have long been considered as farming waste in the rubber plantations without commercial value and gradually become the environmental problem. However, with low ash and high carbon content, RSS becomes potential material for producing activated carbon [10].

The activation process in the production of activated carbon can be done using two methods, i.e., physical and chemical activation. In physical activation, the raw material is carbonized with an oxidizing gas, whereas, in chemical activation, the raw material is impregnated with a chemical reagent. The chemical reagent is required to increase the porosity and surface area of activated carbon; some typical chemical reagents are used, such as potassium hydroxide (KoH), potassium carbonate (K₂CO₃), sodium hydroxide (NaOH), sodium carbonate (Na₂CO₃), phosphoric acid (H₃PO₄), and zinc chloride (ZnCl₂). The second step in chemical activation is the carbonization process. Potassium hydroxide is preferred by some researchers due to high microporosity can be produced [11].

Activated carbon with a higher amorphous percentage will have a higher surface area. In the production process of



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Table 1 Processing parameters in the production of rubber seed shell-derived activated carbon

Parameters	Symbol	Unit	Level 1	Level 2	Level 3
Control factors					
Chemical reagent concentration	A	wt.%	25	50	75
Activation temperature	В	°C	600	700	850
Activation time	C	hours	1.0	2.0	2.5
Response variables					
Product yield	-	wt.%	-	-	-
Amorphous percentage	-	wt.%	-	-	-

activated carbon, the main concerns are obtaining the maximum product yield and amorphous percentage. Many variable affect the product yield and amorphous content, including chemical reagent concentration, activation temperature, and activation time. Typically, the trial and error method with many tests is applied to find out the effect of each variable. However, the optimization of controllable variables can make a significant decrease in the number of experiments. Consequently, the production cost and time loss will be considerably minimized and reduced. As a result, the optimization methods have become the focus of interest for producing the activated carbon.

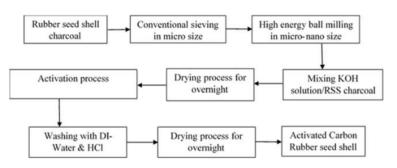
To ptimize the production of activated carbon, the design of experiment methods and statistical analysis have been applied by using the Taguchi method. This method is the most used experimental design method because it provides a vigorous design of experiment (DOE) with a minimum number of experiments. Karna and Sahai [12] provided evidence for applying the Taguchi method to reduce cost and improve quality.

Kundu et al. [13] investigated the activated carbon production from palm kernel shell by controlling four parameters, i.e., microwave power, impregnation ratio (IR), irradiation time, and activating reagent concentration, in four levels. The optimal condition was considered as 800 W of microwave power, 2 of IR, 17 min of irradiation time, and 85 wt.% of phosphoric acid concentration, and the BET (Brunauer–Emmett–Teller) surface area was 1473.55 m²/g. Petuhov [14] also considered similar parameters in the preparation of activated carbons from the walnut shell.

The optimal parameters were found at 450 W of microwave power, 20 min of activation time, 70 wt.% of phosphoric acid concentration, and 0.8–2.0 mm of particle size. The specific surface area and yield of the sample achieved in optimum condition are 918 m² and 39.5%. Particle size and chemical concentration were the most important parameters in optimizing activated c pon production. Syed-Hassan and Zaini [15] prepared activated carbon from the plm kernel shell. The optimized parameter was achieved at the impregnation ratio of 0.55, the activation temperature of 900 °C, and the activation time of 150 min. The highest BET surface area and total pore volume were 1548.0 m²/g and 1.0 cm³, respectively. The impregnation ratio was considered as the main parameter affecting the methane adsorption.

The aim of this research work is to study the effects of process parameters, i.e., chemical reagent concentration, activation temperature, and activation time, on the product yield and amorphous per an intage of activated carbon obtained from rubber seed shell. The Taguchi method with L9 orthogonal array (OA) was applied to design the controllable parameters and response variables for the experiment. The optimal condition for maximizing parameter yield and amorphous percentage of activated carbon was pursued with the signal to noise (S/N) ratio value from the Taguchi method. A significance level of each controllable parameter was statistically examined by ANOVA. Paression analysis was used to predict the measured values. Finally, the comparison process of the results from the experiment and measured values was done for confirmation and validation in this research work.

Fig. 1 Flow diagram for the preparation of rubber seed shell-derived activated carbon





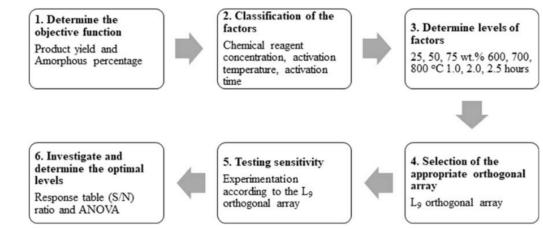


Fig. 2 Preparation of rubber seed shell-derived activated carbon using the Taguchi method

2 Materials and methods

2.1 Materials

In this work, the raw material used for producing activated carbon is RSS collected from a rubber plantation estate located at Bangka Belitung Island province in Indonesia. After washing and drying in the open atmosphere for 3 days before the experiments, the dried RSS was carbonized with a conventional carbonization technique. The carbonized RSS was labeled as RSS charcoal. Two analytical grade chemical reagents, i.e., 85 wt.% potassium hydroxide (KOH) and 0.1 N hydrochloric acid (HCL) purchased from PANREAC AppliChem were then used to proceed with the chemical activation method.

2.2 Preparation of the activated carbons

With high energy impact inside, a planetary ball mill, one of the most rapid techniques to make small particles [16–20], was used to crush the carbonized rubber seed shell. Based on our preliminary experiment results [21], to obtain the smallest particle size of carbon powder, the milling parameters were set to 30 h of milling time and 2:1 of ball mill to powder ratio (BPR). Then, the rubber seed shell charcoals were sieved to 4–5 µm.

According to other researchers' works [22–25], the preparation of activated carbons from rubber seed shell was performed by the chemical activation method with potassium hydroxide as a chemical reagent. Different concentrations of chemical reagent, i.e., 25, 50, and 75 wt.%, were examined.

Table 2 The experimental results and S/N ratios for product yield and amorphous percentage

Test number	Chemical reagent concentration, A (wt.%)	Activation temperature, B (°C)	Activation time, C (h)	Product yield (wt.%)	S/N ratio (dB)	Amorphous percentage (wt.%)	S/N ratio (dB)
1	25	600	1.0	69.4	36.827	92.8	39.3510
2	25	700	2.0	37.94	31.5819	92.9	39.3603
3	25	850	2.5	38.49	31.7070	94.1	39.4718
4	50	600	2.0	53.2	34.5182	92.9	39.3603
5	50	700	2.5	58.8	35.3875	93.4	39.4069
6	50	850	1.0	30.7	29.7428	83.8	38.4649
7	75	600	2.5	51.7	34.2698	93.2	39.3883
8	75	700	1.0	67.3	36.5603	89.7	39.0558
9	75	850	2.0	16.1	24.1365	89.8	39.0655

 T_{Py} , total mean value of product yield = 47.07%; $T_{\mathrm{Py-S/N}}$, total mean value of S/N ratio for product yield = 32.748 dB; T_{Ap} , total mean value of amorphous percentage = 94.1%; $T_{\mathrm{Ap-S/N}}$, total mean value of S/N ratio for amorphous percentage = 39.214 dB



Table 3 Table of response for S/N ratios

Levels	Parameters	Parameters									
	Product yields			Amorphous percentages							
	A (wt.%)	B (°C)	C (h)	A (wt.%)	B (°C)	C (h)					
Level 1	33.37 ^a	35.21 ^a	34.38 ^a	39.39 ^a	39.37 ^a	38.96					
Level 2	33.22	34.51	30.08	39.08	39.27	39.26					
Level 3	31.66	28.53	33.79	39.17	39.00	39.42 ^a					
Delta	1.72	6.68	4.30	0.32	0.37	0.47					
Rank	3	1	2	3	2	1					

a High S/N ratios

During the activation process, 600, 700, and 850 °C working temperatures were tested with a varied time of 1.0, 2.0, and 2.5 h.

The charcoal samples were mixed with KOH solutions using constant impregnation ratio (IR) of 1, which refers to the mass of KOH to the mass of charcoal. For the impregnation process, a hotplate magnetic stirrer was used. The mixture of KOH solution and carbonized RSS was then dehydrated in the oven ovemight at 120 °C. The dried samples were then activated in a muffle furnace at different conditions, determined by the design of experiments, which are shown in Table 1. The activated carbons were gradually cooled down to room temperature, neutralized with a 0.1 M HCl solution and washed with deionized water until the pH value reached between 6 and 7. Finally, the activated carbons were dried at 120 °C overnight. The flow diagram for the activated carbon preparation is shown in Fig. 1.

2.3 Product yield measurement

The product yields of activated carbons were calculated based on the weight of rubber seed shell on a dry basis using Eq. (1) [22, 23].

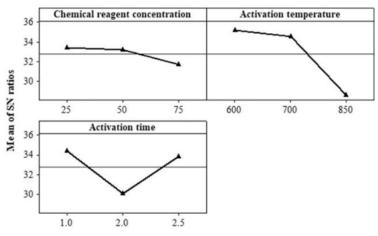
Product yield (wt.%)

$$= \frac{\text{weight of activated carbon}}{\text{weight of rubber seed shell}} x 100 \tag{1}$$

2.4 Amorphous percentage measurement

The amorphous percentage measurement was performed using X-ray diffractometer (D2 PHASER, Bruker) equipped with Cu K α monochromator (λ = 0.154060 nm), 2-theta of

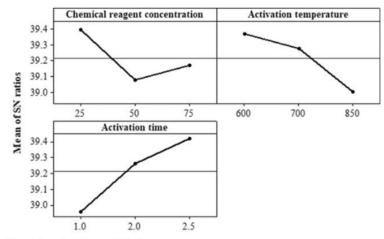
Fig. 3 Main effect plot of S/N ratios for product yield



Signal-to-noise: Larger is better



Fig. 4 Main effect plot of S/N ratios for the amorphous percentage



Signal-to-noise: Larger is better

scanning range varied from 20° to 80° at 0.0326° step size, and 3°/min scanning rate. The voltage supplied was 30 kV with a current of 10 mA. The background method was chosen in order to estimate and analyze the amorphous percentage of activated carbon samples. This method states that the amorphous percentage is represented by the area of the background divided by the total area of the pattern (area under background and the crystalline phase peaks) [24].

$$\frac{S}{N} = -10 \text{ Log}\left(\frac{1}{n}\sum Y^2\right) : \text{The lower-the-better}$$
 (2)

$$\frac{S}{N} = -10 \, \log \left(\frac{1}{n} \sum \frac{1}{Y^2} \right) : \text{The higher-the-better} \tag{3}$$

$$\frac{S}{N} = -10 \, \mathrm{Log} \bigg(\Sigma \frac{Y^2}{Y^2} \bigg) : \text{The nominal--the--better} \tag{4} \label{eq:spectrum}$$

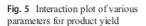
2.5 Signal to noise (S/N) ratio analysis for optimizing multiple factors

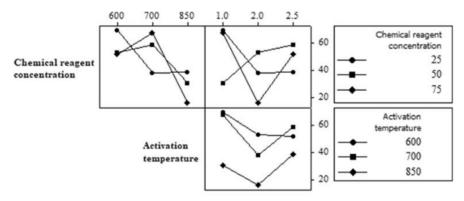
In the Taguchi method, the S/N ratio is used to improve the performance characteristic. Typically, the S/N ratio can be characterized into three categories: the lower-the-better (Eq. (2)), the higher-the-better (Eq. (3)), and the nominal-the-better (Eq. (4)) [25].

where n is the repeated experiment number and Y is the measured value of the response variable.

2.6 Analysis of variance

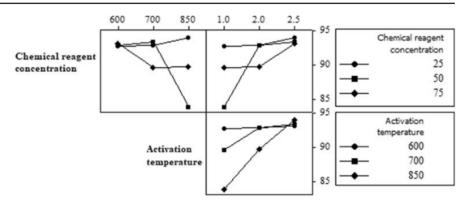
The contribution of each parameter and the most effective parameters are analyzed using the ANOVA approach.





Activation time

Fig. 6 Interaction plot of various parameters for the amorphous percentage



Activation time

3 Design of experiments and optimization

3.1 Experimental design using the Taguchi method

As a simple and efficient approach, the Taguchi method is chosen in this study to optimize the process parameters [26,

27]. The Taguchi method can minimize the test numbers and uncontrolled factors effects by using orthogonal arrays (OA) [28].

The objective of this study is to maximize product yield and amorphous percentage. Therefore, the higher-the-better category for the performance characteristic (S/N ratio) is

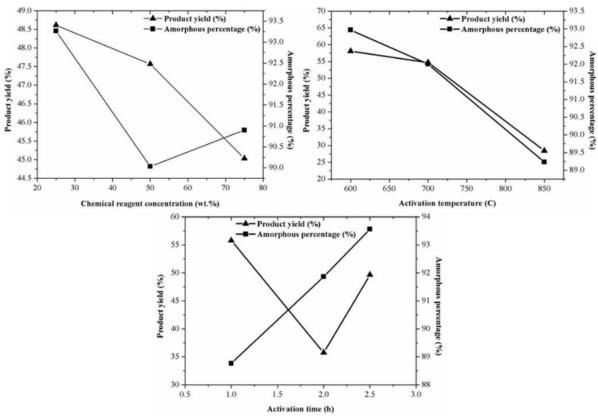


Fig. 7 The effects of process parameters on product yield and amorphous percentage



Table 4 ANOVA results for product yield and amorphous percentage

Variance	DF	SS	MS	F	p	Significance level
Product yield						
A: Chemical reagent concentration	2	20.3	10.1	0.08	0.926	Level 3
B: Activation temperature	2	1581.1	790.5	6.23	0.138	Level 1
C: Activation time	2	633.5	316.7	2.50	0.286	Level 2
Error	2	252.7	126.9	-	-	
Total	8	2488.6	-	-	-	
Amorphous percentag	ge					
A: Chemical reagent concentration	2	16.807	8.403	1.80	0.357	Level 3
B: Activation temperature	2	22.527	11.263	2.42	0.293	Level 2
C: Activation time	2	35.540	17.770	3.81	0.208	Level 1
Error	2	9.327	4.663	-	-	
Total	8	84.200	-	-	-	

DF, degree of freedom; SS, sum of squares; MS, mean squares

chosen. Three controllable factors (chemical reagent concentration for impregnation, temperature for activation, time for activation) at three different levels and two response variables (product yield, amorphous percentage) are considered as parameters shown in Table 1. The approach of applying the Taguchi method to prepare activated carbon is shown in Fig. 2. The procedures consist of determination, classification, investigation, testing, and selection of the orthogonal array for the design of experiments [29].

Table 5 Comparison of deviation values of product yield from the experimental and multiple regression model

Test number	Actual product yield (wt.%)	Predicted product yield (wt.%)	Deviation (wt.%)
1	69.4	69.03	0.37
2	37.94	50.36	12.42
3	38.49	28.725	9.765
4	53.2	60.86	7.66
5	58.8	45.375	13.425
6	30.7	36.48	5.78
7	51.7	55.875	4.175
8	67.3	53.13	14.17
9	16.1	28.31	12.21

Table 6 Comparison of deviation values of amorphous percentage from the experimental and multiple regression model

Test number	Actual amorphous percentage (wt.%)	Predicted amorphous percentage (wt.%)	Deviation (wt.%)
1	92.8	91.6875	1.1125
2	92.9	93.3575	0.4575
3	94.1	92.6725	1.4275
4	92.9	93.695	0.795
5	93.4	93.77	0.37
6	83.8	86.705	2.905
7	93.2	94.1075	0.9075
8	89.7	87.8025	1.8975
9	89.8	88.7125	1.0875

4 Analysis and evaluation of experimental results

4.1 Signal to noise (S/N) ratio

The effects of controllable factors on the product yield and amorphous percentage are represented by the S/N ratio. Table 2 shows the results of S/N ratios for the product yield and amorphous percentage. The total mean values of product yield ($T_{\rm Py}$) and amorphous percentage ($T_{\rm Ap}$) resulted from varied activation processes are 47.07% and 91.4% respectively, while the total mean values of S/N ratios for product yield ($T_{\rm Py-S/N} = 32.75$ dB) and amorphous percentage ($T_{\rm Ap-S/N} = 39.21$ dB) are calculated accordingly.

The responses of S/N ratios are summarized in Table 3. For each parameter, the factor level with the highest S/N ratio indicates the best level to optimize the response. Hence, the maximum product yield will be achieved at level 1 of each control factor, i.e., 25 wt.% of chemical reagent concentration,

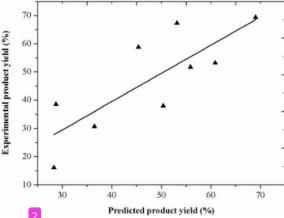


Fig. 8 Comparison of the experimental results and predicted values for product yield

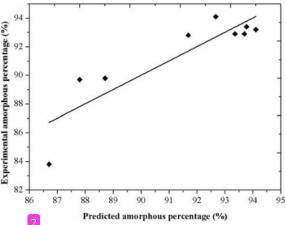


Fig. 9 Comparison of the experimental results and predicted values for amorphous percentage

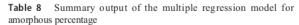
600 °C of activation temperature and 1.0 h of activation time. The S/N ratios for each pagneter are 33.37, 35.21, and 34.38 dB, respectively. The main effects plot of S/N ratios for product yield and amorphous percentage are shown in Figs. 3 and 4. The optimal level for maximum amorphous percentage is achieved in level 1 of chemical reagent concentration (25 wt.%) and activation temperature (600 °C), and level 3 of activation time (2.5 h). The highest S/N ratios for optimal parameters are 39.39, 39.37, and 39.42.

Interaction of control factors for product yield and amorphous percentage are shown in Figs. 5 and 6. Parallel lines mean no mutual effect while crossing lines indicate significant interaction. In Fig. 5, there are interactions between three chemical reagent concentrations and three activation temperatures.

Figure 6 presents the interaction plot for the amorphous percentage. The chemical reagent concentration of 25 wt.% interacts with 50 wt.% at 600 and 700 °C, and with 75 wt.% at 610 °C. Besides, 50 and 75 wt.% interact at activation temperatures of 600 and 775 °C. The chemical reagent concentration of 25 wt.% interacts with 50 wt.% at the activation time of 2.0 h. On the other hand, the chemical reagent concentrations of 50 and 75 wt.% interact at activation times of 1.8 and 2.5 h.

Table 7 Summary output of multiple regression model for product yield

Predictor	Coef	SE Coef	T statistic	p value
Constant	150.57	35.60	4.23	0.08
Chemical reagent concentration	-0.0715	0.2175	-0.33	0.756
Activation temperature Activation time	-0.12313 -6.371	0.04322 7.120	- 2.85 - 0.89	0.036 0.412



Predictor	Coef	SE Coef	T statistic	p value
Constant	98.827	5.113	19.33	0.000
Chemical reagent concentration	-0.04733	0.03124	-1.52	0.190
Activation temperature	-0.015211	0.006207	-2.45	0.058
Activation time	3.186	1.023	3.12	0.026

The interaction plot shows no interaction between chemical reagent concentration of 25 and 75 wt.%. The activation temperature of 600 and 700 °C interact at the activation time of 2.0 h, and with the activation temperature of 850 °C at the activation time of 2.3 h. The activation temperature of 700 and 850 °C interacts at the activation time of 2.4 h.

Looking at each factor in these plots, the significant interactions are found in more nonparallel lines. The more nonparallel lines mean the greater strength of the interaction. There are significant interactions between chemical reagent concentration and activation temperature, while activation time shows less interaction with the factors. As a conclusion based on the interaction plot, chemical reagent concentration and activation temperature are important parameters in the activation process.

4.2 Experimental results analysis

The effects of chemical reagent 1 heentration, activation temperature, and activation time on product yield and amorphous percentage are shown in Fig. 7. Activation with 25 wt.% concentration KOH results in the highest product yield and amorphous percentage. At each chemical reagent concentration, the product yield of activated carbon linearly decreased with the chemical reagent at higher concentrations. The higher the concentration, the lower the product yield. The lowest amorphous percentage is reached at 50 wt.% of chemical reagent concentration. The highest product yield and amorphous percentage are obtained with the lowest activation temperature. Increasing the activation temperature will decrease the

Table 9 ANOVA for testing significance of regression model for product yield

Source	Degree of freedom	Sum of squares	Mean square	f_0	p value
Regression	3	1601.5	533.8	3.01	0.133
Residual error	5	887.1	177.4		
Total	8	2488.6			





Table 10 ANOVA for testing significance of the regression model for amorphous percentage

Source	Degree of freedom	Sum of squares	Mean square	f_0	p value
Regression	3	65.902	21.967	6.00	0.041
Residual error	5	18.298	3.660		
Total	8	84.200			

product yield and amorphous percentage. The longer the activation time, the higher the amorphous percentage, whereas the highest product yield results from the lowest activation time.

4.3 Analysis of variance

The ANOVA is a statistical method used for finding the significance degree of dependent factors that affect the performance. The ANOVA results for the product yield and the amorphous percentage are summarized in Table 4. The results show that, for product yield, the F value of the activation temperature is larger than chemical reagent concentration and activation time; for amorphous percentage, the activation time is larger than chemical reagent concentration and activation temperature. Therefore, the activation temperature (factor B with an F value of 6.23) has a strong effect on the product yield, whereas the activation time (factor C with an F value of 3.81) has a strong effect on the amorphous percentage. The higher the F value, the higher the significance degree. In other words, a factor with higher F value affects more on the performance [30].

4.4 Regression analysis of product yield and amorphous percentage

A multiple regression model was studied to examine the interaction between controllable parameters (or dependent variables) and uncontrollable parameters (or dependent variables). The independent variables were chemical reagent

concentration, activation temperature, and activation time, while the productive equations obtained from the multiple regression model of product yield and amorphous percentage are shown in Eqs. (5) and (6).

$$P_{y,Predicted} = 151 - 0.072A - 0.123B - 6.37C$$
 (5)

$$A_{\text{p.Predicted}} = 98.8 - 0.0473A - 0.0152B + 3.19C \tag{6}$$

where $P_{y, \text{ Predicted}}$ is the predictive product yield (in wt.%), $A_{p, \text{ Predicted}}$ is the predictive amorphous percentage (in wt.%), A is the chemical reagent concentration (in wt.%), B is the activation temperature (in °C), and C is the activation time (in hours).

The developed multiple regression models (Eqs. (5) and (6)) are verified and validated by comparing the values of product yield and amorphous percentage obtained via an experiment with the predicted output of the multiple regression model (Tables 5 and 6).

The comparisons between experimental results and predicted results obtained by the multiple regression model are given in Figs. 8 and 9. The experimental results function as actual values and the multiple regression model results are considered as the predicted values. The R-squared (R^2) values obtained from the multiple regression models of product yield and amorphous percentage are 64.4% and 78.3% respectively.

In Table 7, the regression putputs for product yield are summarized. The p value of chemical reagent concentration, activation temperature, and activation time are 0.756, 0.036,

Table 11 Mean response table of product yield and amorphous percentage

Levels	Control factors									
	Product yield			Amorphous percentage						
	8 A	В	C	A	В	C				
Level 1	48.61 ^a	58.10 ^a	55.80 ^a	93.27ª	92.97ª	88.77				
Level 2	47.57	54.68	35.75	90.03	92.00	91.87				
Level 3	45.03	28.43	49.66	90.90	89.23	93.57a				
Delta	3.58	29.67	20.05	3.23	3.73	4.80				
Rank	3	1	2	3	2	1				

a Optimum level



Table 12 Experimental results and predicted values for optimal product yield

	Taguchi method			Multiple regression model			
	Prediction	Confirmation experiment	Error (%)	Prediction	Confirmation experiment	Error (%)	
Level	$A_1B_1C_1$	$A_1B_1C_1$	-	$A_1B_1C_1$	$A_1B_1C_1$	-	
Product yield (wt.%)	68.37	66.06	3.38	69.03	66.06	4.30	

and 0.412, respectively. The p value for activation temperature (0.036) is lower than the common alpha level of 0.05, which indicates that the activation temperature is statistically significant, while the p value of chemical reagent concentration (0.756) and activation time (0.412) is greater than the common alpha level of 0.05, which mean both of them are statistically insignificant.

Table 8 shows the regression output for the amorphous percentage. The p value of chemical reagent concentration, activation temperature, and activation time are 0.190, 0.058, and 0.026, respectively. The predictor variables of activation temperature (0.058) and activation time (0.026) are significant because both p values are lefter than or equal to the common alpha level of 0.05, while the p value of chemical reagent concentration (0.190) is greater than the common alpha level of 0.05, which means it is not statistically significant. A low p value (<0.05) means the null hypothesis is rejected. In other words, a predictor that has a low p value is likely a meaningful addition to the model, because changes in the predictor's value are related to changes in the response variable. Conversely, a larger (insignificant) p value suggests that changes in the predictor are not associated with changes in the response.

The test of hypotheses is applied to determine whether multiple relationships exist between the response variable and the regressor variables. For the test of hypotheses, the test for significance of the regression is done. Tables 9 and 10 present ANOVA for testing the significance of regression in multiple regression. The test intends to determine whether a linear relationship exists between the response variable (i.e., product yield and amorphous percentage) and a subset of the regressor variables (i.e., chemical reagent concentration, activation temperature, and activation time). The appropriate hypothesis is H_0 : $\beta 1 = \beta 2 = \beta 3 = 0$. The testing significance

shows that the p value for product yield is considerably higher than $\alpha=0.05$, while the p value for the amorphous percentage is smaller than $\alpha=0.05$. For the product yield, p value > 0.05 indicates the failure of rejecting the null hypothesis (H_0), whereas, for the amorphous percentage, the null hypothesis (H_0) is rejected due to p value < 0.05, which concludes that at least one of the variables contributes significantly to the multiple regression model. This result confirms that activation temperature and activation time contribute significantly to the model for amorphous percentage.

4.5 Estimation of the optimal product yield and amorphous percentage

The optimization recess in the Taguchi method has been conducted. To gain the optimal product yield and amorphous percentage, Eqs. (7) and (8) are applied.

$$P_{y,\text{Optimal}} = (A_1 - T_{py}) + (B_1 - T_{py}) + (C_1 - T_{py}) + T_{py}$$
 (7)

$$A_{p,Optimal} = (A_1 - T_{Ap}) + (B_1 - T_{Ap}) + (C_3 - T_{Ap}) + T_{Ap}$$
 (8)

where $P_{\rm y,\ Optimal}$ is the optimal product yield (in wt.%), $A_{\rm p,\ Optimal}$ is the optimal amorphous percentage (in wt.%), $T_{\rm py}$ is the total mean value for product yield, and $T_{\rm Ap}$ is the total mean value for amorphous percentage. The levels of process parameters in producing optimal product yield and amorphous percentage are represented in $(A_1,\ B_1,\ C_1)$ and $(A_1,\ B_1,\ C_3)$, respectively. Table 11 shows the means of response characteristics for product yield and amorphous percentage. The 12 phest mean values indicate the most optimal conditions. The optimal product yield and amorphous percentage are 68.37% and 91.61% respectively.

Table 13 Experimental results and predicted values for optimal amorphous percentage

	Taguchi method			Multiple regression model		
	Prediction	Confirmation experiment	Error (%)	Prediction	Confirmation experiment	Error (%)
Level	$A_1B_1C_3$	$A_1B_1C_3$	-	$A_1B_1C_3$	$A_1B_1C_3$	-
Amorphous percentage (wt.%)	91.61	85.7	6.45	96.47	85.7	11.16



Table 14 Experimental results and predicted values for random condition

	Taguchi method			Multiple regression model		
	Prediction	Confirmation experiment	Error (%)	Prediction	Confirmation experiment	Error (%)
Level	$A_3B_2C_1$	$A_3B_2C_1$	-	$A_3B_2C_1$	$A_3B_2C_1$	-
Product yield (wt.%)	61.37	67.3	8.81	53.13	67.3	21.05
Amorphous percentage (wt.%)	83.47	89.7	6.95	87.80	89.7	2.12

4.6 Confirmation test

The confirmation test of the process parameters is made for the Taguchi method and multiple regression equations at optimal levels 21 Tables 12, 13, and 14, the comparisons of the experiment results and the predicted values are obtained by 2 ing the Taguchi method and multiple regression equations. The predicted and the experimental values are very close to each other. The percentages of errors are 3.38 and 6.45% for the Taguchi method and 4.30 and 11.16% for the multiple regres 3 n model. All percentages of errors are lower than 20%. For reliable statistical analyses, the percentage of error 3 lues for optimization must be within 20% [31]. Therefore, the confirmation test proofs that the Taguchi Method and the multiple regression model are effective for optimization.

5 Conclusion

This study has investigated the optimal process parameters of activated carbon production obtained from rubber seed shells by using the Tague method. Three important parameters as control factors, i.e., chemical reagent concentration, activation temperature, and activation time, have been studied. By applying ANOVA, the experimental data are analyzed. The product yield of activated carbon declines with increasing chemical reagent concentration and activation temperature, while the amorphous percentage declines with increasing activation temperature and increases with increasing activation time.

The optimal conditions to reach maximum product yield are $A_1B_1C_1$ (25 wt.% of chemical reagent concentration, 600 °C of temperature, and 1 h of activation time). To obtain maximum amorphous percentage, the optimal conditions are $A_1B_1C_3$ (25 wt.% of chemical reagent concentration, 600 °C of activation temperature, and 2.5 h of activation time).

The activation temperature influences the product yield most, and activation time affects strongly on amorphous percentage. In the confirmation test, the developed multiple regression equation and experimental values present a low percentage of error (<20%) to indicate the accuracy. This study has provided quality evidences that the Taguchi method is an

applicable and effective methodology with precise results for optimizing the parameters in the activated carbon production.

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