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Hydrothermal Synthesis of Well-Crystallized Boehmite from Lapindo Mud: Process Optimization

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Abstract

A well-crystallized form of boehmite was successfully synthesized from alumina derived from Lapindo Mud using hydrothermal methods. A comprehensive evaluation and analysis of the reaction temperature, reaction time, and pH of the hydrothermal system was carried out with the aim to determine the most suitable variables for optimization boehmite production. For the purpose of evaluating the model, analyzing the data, and optimizing the operating conditions, Central Composite Design (CCD) was employed alongside the statistical programs Statistica 6.0. The results indicated that the optimal conditions are a pH of 9.1, a reaction temperature of 191°C, and a reaction time of 20 hours and 12 minutes. A high yield of 98.49% has been achieved by synthesizing boehmite under optimum operating conditions, according to the results. The sample that had been synthesized under optimum operating conditions was employed for evaluating the results. Using X-Ray Diffraction (XRD) and Fourier Transform Infrared Spectroscopy (FTIR), the average particle size, crystallinity index, and functional groups of the synthesized boehmite have been identified. The FTIR analysis showed that the functional groups present in the synthesized boehmite closely resemble those found in commercial boehmite. This similarity in functional groups indicates that the chemical composition and bonding characteristics of the synthesized boehmite are consistent with those of the commercially available product. The synthesized material's excellent quality was highlighted by the XRD result, which is showed a high crystallinity index and a clearly defined average particle size. This synthesis process successfully synthesized boehmite with desirable properties using alumina from Lapindo Mud.

Keywords: Alumina,Boehmite, Hydrothermal, Optimization

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1. Introduction

Energy is a basic necessity nowadays. Almost all aspects of life require energy. So far, energy fulfillment is still supported by natural oil. The hydrocarbon components in natural oil need to be broken down to obtain derivatives with the desired specifications. Catalysts assist the breaking of hydrocarbon chains in natural oil. However, each catalyst does not immediately meet the specifications suitable for the process. Therefore, the process of adding other components that can cover the shortcomings of the catalyst can be called a supported-catalyst. Boehmite is one type of material that is often used as a catalyst buffer, for example in multicomponent, hydrogenation, oxidation, epoxidation, and cross-linking reactions in many types of hydrocarbons [1]. Several researchers have investigated the process of synthesizing boehmite from various sources. Hydrothermal, solvo thermal, and sol-gel methods are methods that can be used to synthesize boehmite [2-4].

The hydrothermal method is the best method to synthesize boehmite based on consideration of several aspects. The hydrothermal method uses water as a solvent in the process. The use of water is considered to reduce hazardous waste from the synthesis process. In addition, the purification process of the resulting boehmite is easier than the solvothermal method, which uses non-aqueous solvents such as alcohol. The hydrothermal method also has a lower reaction time than the sol-gel method. Boehmite is available in nature in amorphous form with many impurities in the bauxite mineral matrix which also contains Al-hydroxide, kaolinite, Fe-oxy hydroxide and Ti-oxide [5] and from other natural sources. Alumina is a material that can be extracted from bauxite and other natural sources with higher purity. Alumina can be used as a catalyst or as a reagent for other materials, one of which is boehmite. The Al-O bond in alumina will be broken by the addition of -OH groups in the hydroxylation process.

One other source that contains alumina in high quantities is Lapindo mud. Lapindo mud is a problem that has yet to be solved. According to geologists, these eruptions are expected to last for at least 30 years and are related to volcanic activity [6]. Lapindo mud contains a lot minerals, such as silica (45%) iron oxide (24.1%), alumina (14%), calcium oxide as (6.1%), molybdenum oxide (4.6%), potassium oxide (2.8%), and titanium dioxide (1.9%) [7].This research studies the optimum conditions of the variables that affect the synthesis which is pH, reaction temperature, and reaction time in the formation of boehmite. The study was carried out using Response Surface Methodology (RSM), which is a statistical and mathematical technique to model and analyze data to obtain optimum conditions. This research is expected to contribute to the unutilized Lapindo mud waste processing sector. In addition, to provide the needs of alumina and boehmite from other sources.

2. Materials and Methods

2.1. Material

Lapindo mud was collected from Sidoarjo, East Java, Indonesia. Sulfuric acid was purchased from Supelco. Sodium hydroxide was purchased fron Merck. Hydrochloric acid was purchased from Supelco.

2.2. Method

2.2.1. Extraction of alumina

The alumina extraction process from Lapindo mud was carried out according to Boudreault et al. [8] with slight modifications. Lapindo mud was dried and sieved to a size of 100 mesh. The extraction process was carried out with the reflux method in a 6 M sulfuric acid solution (1:4 w/v) under stirring conditions at 80ºC for 5 hours. The solution was filtered to separate the residue and filtrate. Filtrate was used for further selective precipitation under pH 12 with the addition of 6M sodium hydroxide. The process was carried out under stirring conditions at 80ºC for 3 hours to precipitate Fe^{3+}. The mixture was filtered. The filtrate was added by 6M of hydrochloric acid until it reached pH 8. The precipitation process was performed under stirring conditions at 80ºC fot 3 hours. The solution was filtered. The precipitate was washed with distilled water and dried until it reached a constant mass. The aluminum hydroxide was calcinated at 600ºC for 4 hours.

2.2.2. Synthesis of boehmite

The synthesis of boehmite was performed with hydrothermal method according to Kozerozhets et al. [9]with a slight modification. Alumina from extraction process used as a reagent and dissolved with the distilled water and an adjusted pH by hydrochloric acid and sodium hydroxide. The hydrothermal method was performed in a Teflon with a filling factor of 80%. The synthesized boehmite filtered and washed with distillated water and dried overnight. The boehmite was studied with Fourier Transform-Infrared (FTIR) and X-Ray Diffraction (XRD).

2.2.3. Optimization

The hydrothermal synthesis of boehmite was optimized with response surface methodology (RSM) and central composite design (CCD). The independent variables evaluated are pH (X_1), reaction temperature (X_2), and reaction time (X_3). The independent variables were set at pH 4-12, reaction temperature of 170-200ºC, and reaction time of 16–24 hours. The yield of the product was chosen as the response for the evaluation factor. The real independent variables were translated into coded variables (-1 min, +1 max) in order to evaluate the response surface approach. Table 1 shows the independent's level for the synthesis process. Each response was used to construct a mathematical model that uses a second-order polynomial in order to determine a critical point (minimum, maximum, or saddle), as shown in Eq. (1):

$$Y = \beta_0 + \sum_i^n \beta_i X_i + \sum_i^n \beta_{ii} X_i^2 + + \sum_i^n \sum_j^n \beta_{ij} X_i X_j + \varepsilon \qquad (1)$$

Y is the yield of product; β_0 is the constant coefficient; β_i is the linear coefficients; β_{ij} is the interaction coefficients; β_{ii} is the quadratic coefficients; and X_i, X_j are the coded values of the experimental variables. In this study, the independent variables investigated were: pH (X_1), reaction temperature (X_2), and reaction time (X_3), with Y, yield of the product, set as the response variable. Therefore, Equation 1 can be rewritten as Equation 2:

$$Y = \beta_0+\beta_1X_1+ \beta_2X_2+ \beta_3X_3+ \beta_1X_1^2+ \beta_2X_2^2+ \beta_3X_3^2 +\beta_{12}X_1 X_2+ \beta_{13}X_1 X_3+ \beta_{23}X_2 X_3 \qquad (2)$$

Regression were used to determine the operating condition to synthesis boehmite with hydrothermal method using Statistica 6.0 software. Additional test were carried out after the RSM optimization to confirm and verify the validity of the established model equation as well as the statistic experimental design. The software made it possible to create a graph of surface and countour response value in a three-dimensional plot. The confident level for comparing mean total yield in multiple experimental runs was set at p=0.05 or a significant level of 95%.

3. Results and discussion

3.1. Development of the statistic mode

The fitness of the model is tested to determine the scope of the model in representing the independent variable on the dependent variable, which can be seen from the determination value and the lack of fit test, which is explained by the analysis of variance (ANOVA). Table 2 presents the results from the Boehmite optimization parameter experimental response analysis and response prediction. The boehmite yield, which was collected from the optimization process experiment by sampling 16 times with multiple combinations of independent variables, exhibits significant results in relation to the model's projected value. The difference between experimental boehmite yield and model boehmite yield is less than 0.05, which indicates the model can predict the optimization results. Regression and the coefficient of determination will be discussed in more detail later on. These results can also be illustrated by the prediction vs actual graph of boehmite yield obtained by the optimization process. The point distribution of each experimental run is close to the diagonal line. Points that are near the diagonal line suggest that there is a similarity in the yield boehmite value between the experimental and model-predicted values.

Analysis of Variance (ANOVA) was conducted as a statistical technique that can interpret experimental results by looking at the contribution ratio of each parameter. ANOVA is used to see the significance of each parameter to the overall model [10]. The results of the ANOVA test are shown in

Table 3. Simultaneous regression analysis was conducted with a significance limit of $\alpha = 0.05$ or 5%. The ANOVA test results showed that the interaction between temperature and time had no significant effect on the response ($P > 0.05$). This hypothesis can be validated with the F-test (Fischer-Test). Based on the F-value analysis on the interaction between temperature and time, the F-value < F-table (1.02 < 3.388), the parameter has no significant effect on the dependent variable, H_0 is accepted, H_1 is rejected. The F-value analysis of the model shows F-value > F-table (217.99 > 5.318), H_0 is rejected, H_1 is accepted. Based on the ANOVA test, it can be concluded that the independent variables, namely pH, reaction temperature, and reaction time, as well as the interaction between pH with reaction temperature and pH with reaction time, have an effect on the model.

The lack of fit test is carried out to explain the inaccuracy of the model used. Based on the boehmite optimization results described in the ANOVA table, the lack of fit value obtained is 0.7421. The lack of fit value obtained is greater than the significance limit (0.7421 > 0.05). This indicates that there is no lack of fit in the model. The model used is able to predict the operating conditions in the boehmite synthesis process using the hydrothermal method to obtain the optimum yield. The coefficient of determination (R^2) explains the amount of variance in the dependent variable that can be explained by the independent variables. The R^2 value varies between $-\infty$ to 1 where the closer the value is to 1, the better the data is represented by the model [11]. Optimization of the boehmite synthesis process with the hydrothermal method resulted in a determination value of R^2 0.9964. This shows that the model can represent 99.64% of the data. The R^2_{adj} value of 0.9924 indicates that 99.24% of the experimental data is in agreement with the data predicted by the model. The significance of each independent variable can also be explained with a Pareto Chart.

Pareto charts are presented as bar charts that can define the significance of each independent variable and observe its effects on the response analysis used [12]. Bars that cross the red dashed line declared to have a significant effect on yield, while bars that do not cross the red dashed line are considered to have no effect on the yield of the model. The Pareto chart for the model in the boehmite optimization process is presented in Figure 2. The results of testing the significance of the independent variables used in the Pareto chart show that the independent variables of pH, reaction temperature, and reaction time in both linear and quadratic forms have a significant effect on the response of the model. The interaction between X_1 and X_2 and X_1 and X_3 has a significant influence on the response. The interaction between X_2 and X_3 is the only variant that does not have a significant effect on the response of the model so this term can be ignored. A mathematical model that shows the effect of the independent variables, pH, reaction temperature, and reaction time, can be developed after evaluating the regression coefficients on the boehmite yield response. The mathematical model is determined as follows:

$$Y = -0.9644+0.8410X_1+ 0.7773X_2+ 2.1131X_3 - 0.0579X_1^2 -0.0021X_2^2- 0.0503X_3^2+0.0021X_1 X_2- 0.0090X_1 X_3 \quad (3)$$

3.2. Optimum condition of boehmite synthesis process

The optimum condition of the optimization can be described by response surface curves. The response profile to the independent variable may be seen on the surface response curve; the closer the reaction is to the ideal combination of independent variables, the redder the curve area. The surface response curve is drawn in the form of three-dimensional contour plots based on independent variables namely pH, reaction temperature, and reaction time. The response surface graph is presented in Figure 3. The highest response results can be seen from the response surface plot where the highest boehmite yield is at operating conditions of pH 8-10, reaction temperature 180-200°C, and reaction time 19-21 hours. Zhang et al. [13] showed that in the pH range of ~10, boehmite has a three-dimensional hexagonal shape. Zhang et al. [14] also reported that boehmite synthesized at a temperature of 200 and a time of 24 hours has a shape that tends to be three-dimensional (3D). This allows a small possibility that boehmite particles can pass through the filtration process to maximize yield results. Kozerozhets et al. [9] also stated that reaction temperatures below 150°C could not convert alumina into boehmite perfectly in the time observed. It can be summarized that increasing the reaction temperature in boehmite synthesis can increase the yield rate of boehmite. However, the application of excessive temperature can further convert boehmite into other compounds. According to the Handbook of TGA [15], at temperatures above 230°C, boehmite will hydroxylate into λ-Al_2O_3 which is not the desired product. The critical values, which are the optimum conditions of the independent variables predicted by the system, shown in Table 4.6. The model predicted that the yield of boehmite at optimum conditions is 98, 45%. The validation process was carried out on the boehmite synthesis process at the optimum operating conditions according to the model. The result obtained was 98.49%. This shows that the model successfully represents the experimental results.

3.3. Characterization of synthesized boehmite

Boehmite that has synthesized by the hydrothermal method is then analyzed for product characteristics. The samples analyzed were samples with optimum operating variables. Boehmite characterization carried out using Fourier Transform Infra-Red (FT-IR) spectroscopy and X-Ray Diffraction (XRD) instruments. Boehmite analysis is characterized by stretching and bending of the functional group which is clearly related to the interlayer bonding of the structure [16]. The results of FT-IR analysis showed that the transmittance peaks between the synthesized boehmite and commercial boehmite were relatively similar despite the differences in the intensity of some peaks according to FT-IR analysis. Wavelengths of 735 cm^{-1}, 739 cm^{-1}, 1073 cm-1, 3090 cm^{-1}, 3287 cm^{-1}, and 3295 cm^{-1} are characteristic of boehmite functional groups [17]. The wavelengths 735 cm^{-1} and 739 cm^{-1} indicate the presence of symmetric vibrations in the Al-O group associated with Al-O-Al vibrations of distorted AlO_6 [16]. The wavelength of 1073 cm^{-1} indicates the symmetric deformation of the Al-O-H group [18-19].

Transmittance at wavelengths of 3090 cm^{-1}, 3287 cm^{-1}, and 3295 cm^{-1} indicates the presence of strain vibrations of the -OH group in boehmite. The wider curve in the spectrum of synthesized boehmite can occur due to the presence of water molecules trapped in particles that do not react with aluminum [20]. The weak intensity at a wavelength of 1641 cm^{-1} indicates stretching vibrations of H-O-H groups associated with adsorbed water molecules [2-3-21].

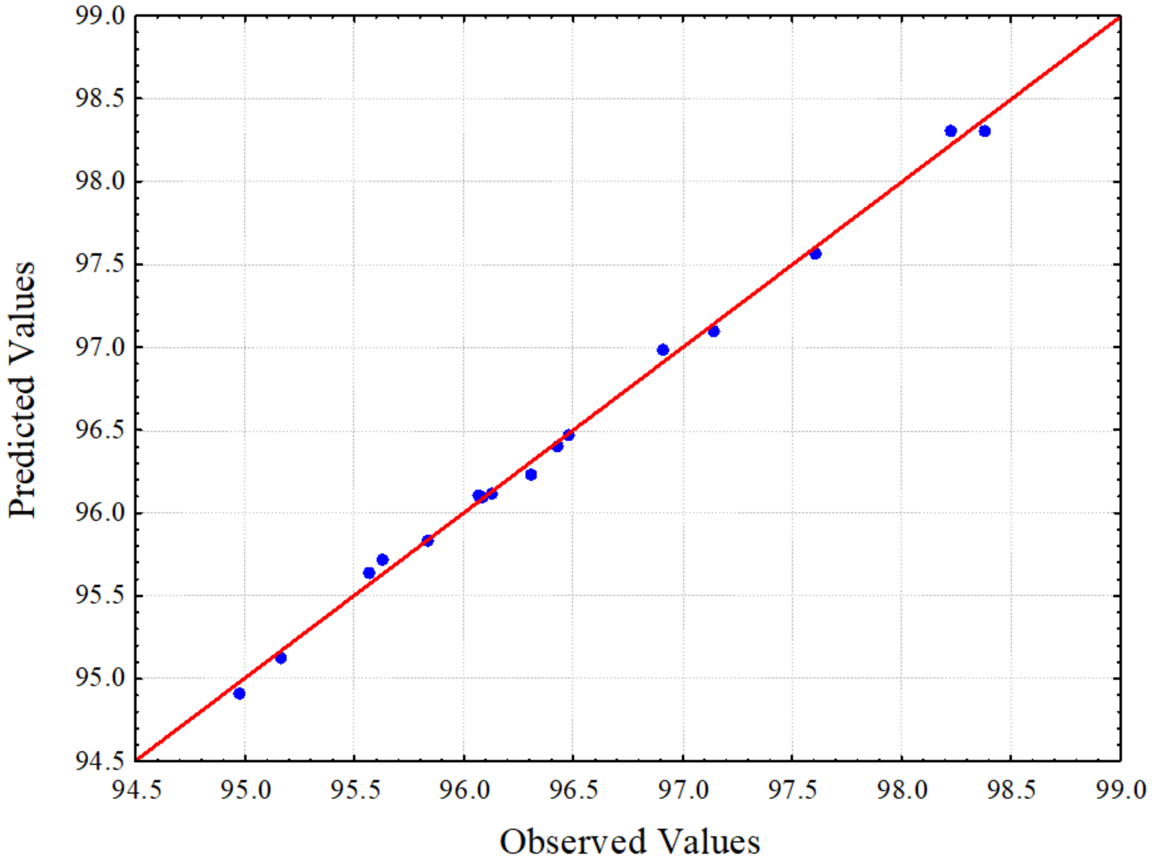

Figure 1: Predicted vs observed values of the model

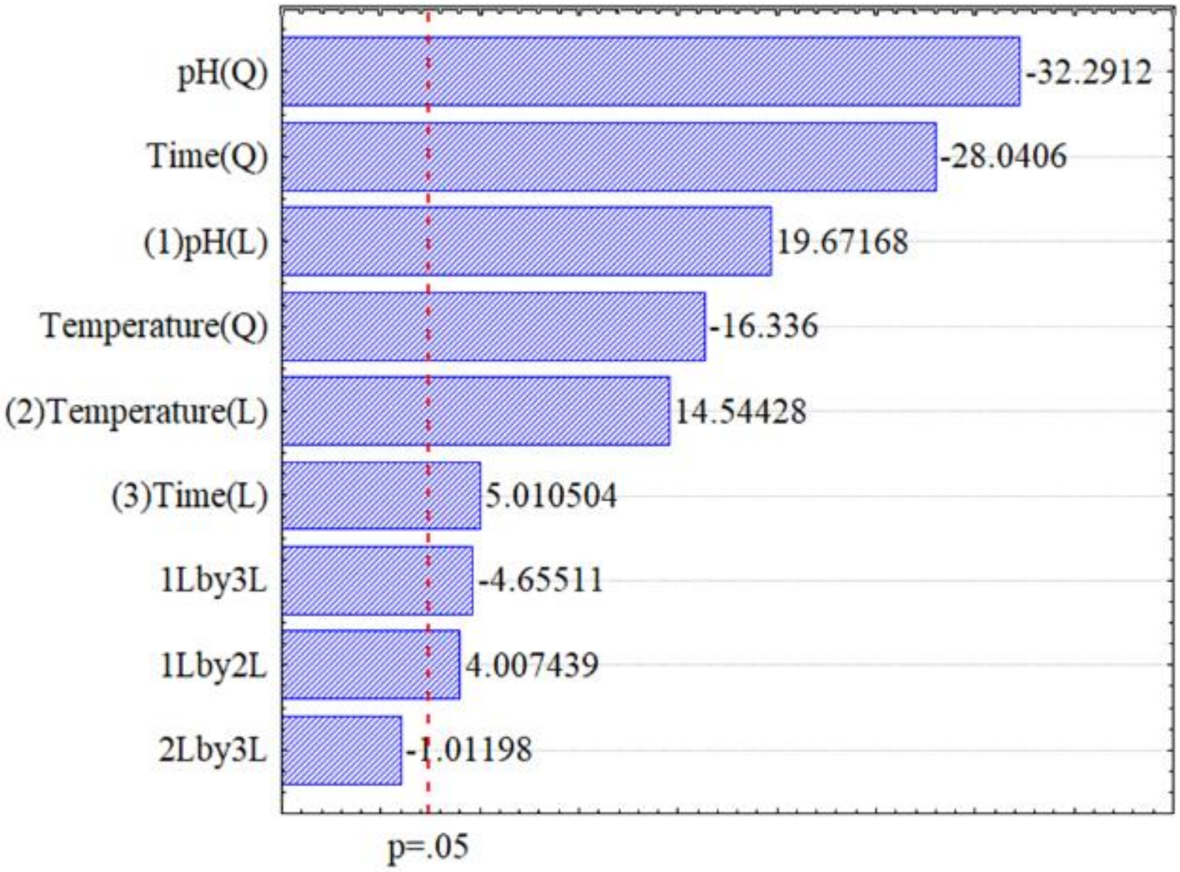

Figure 2: Pareto chart of the observed operating condition of boehmite's synthesis process

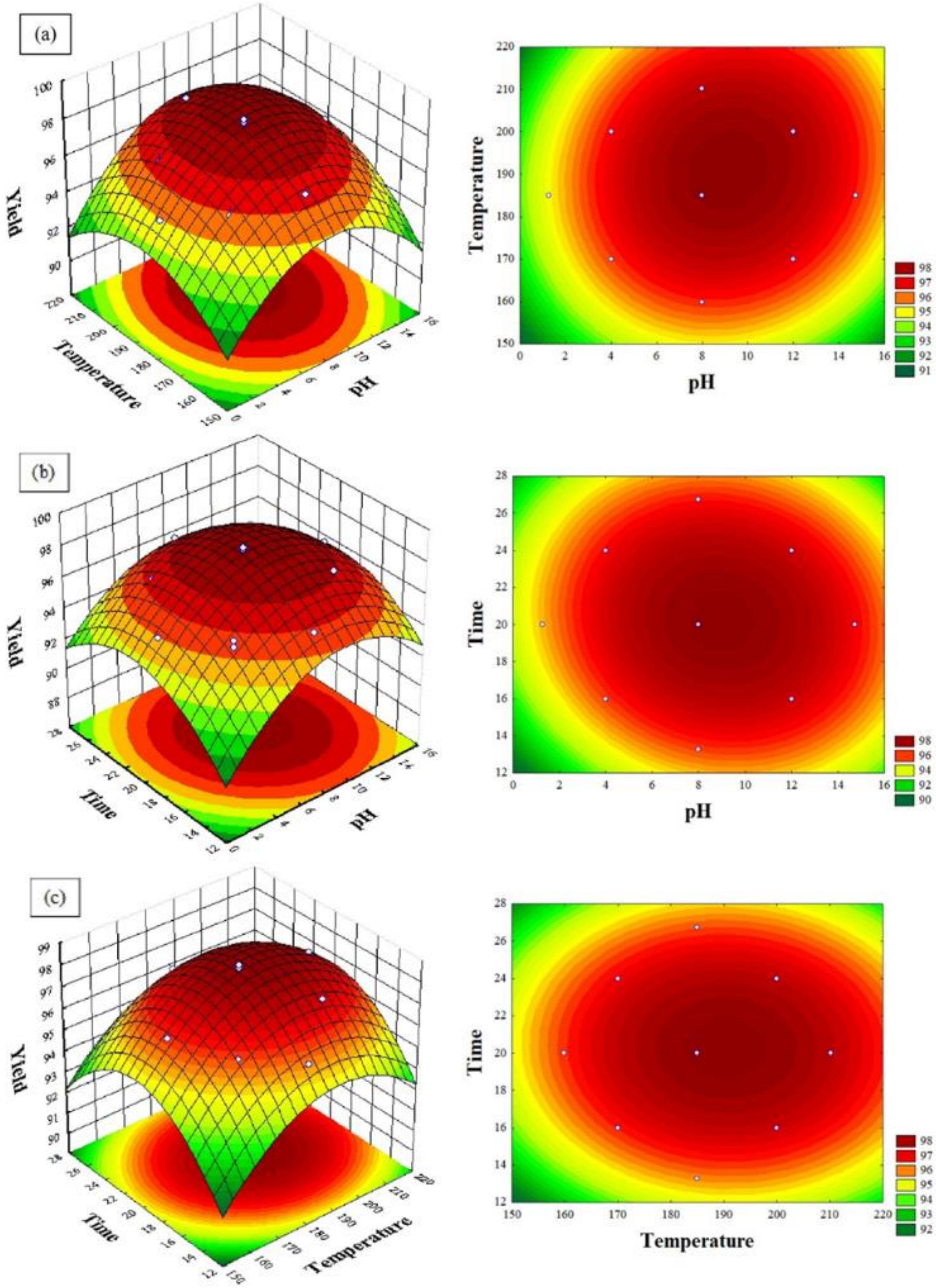

Figure 3: Three-dimensional surface (3D) and contour plot of the interaction of: (a) pH and reaction temperature; (b) pH and reaction time; and (c) reaction temperature and reaction time

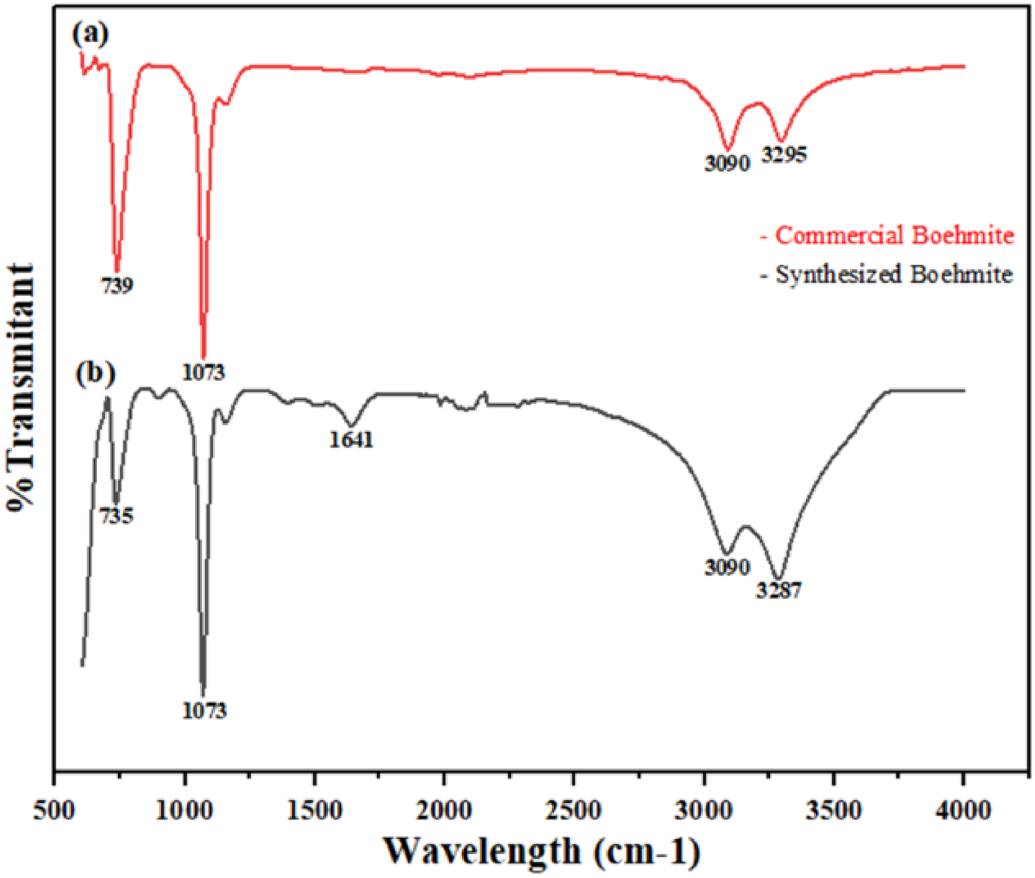

Figure 4: FTIR analyze of boehmite

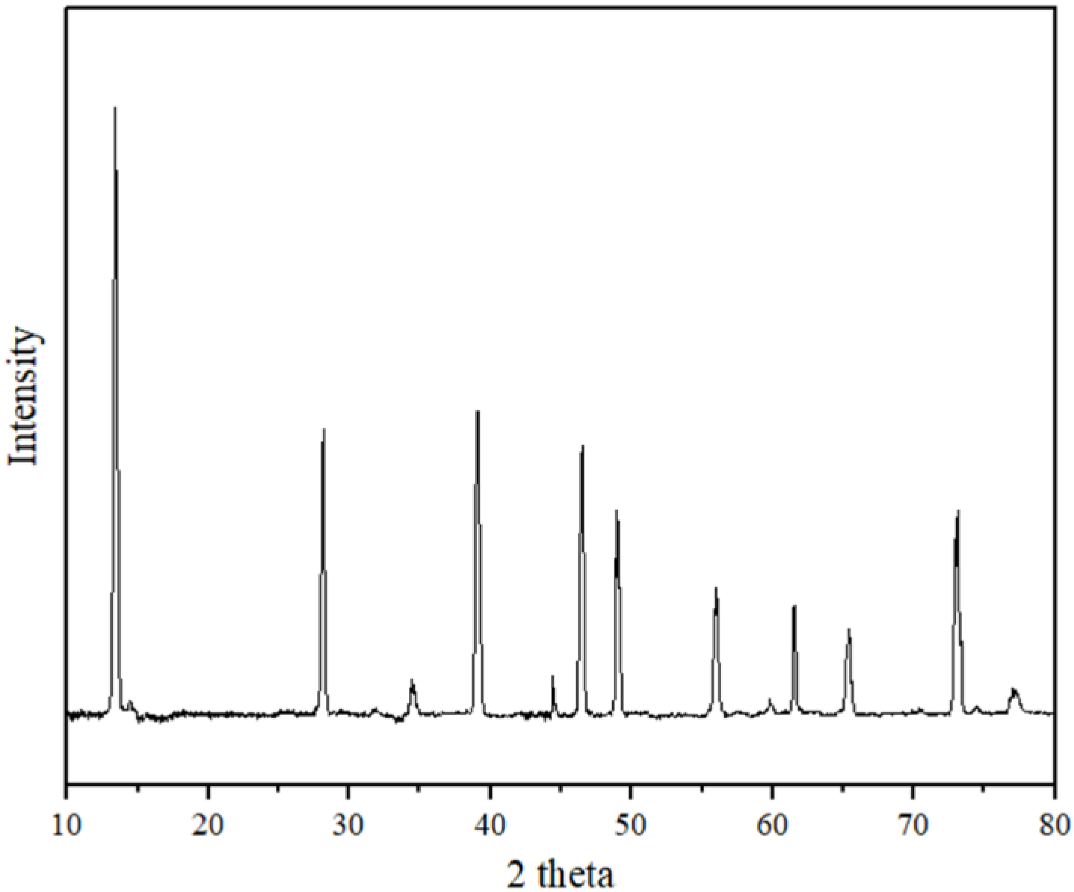

Figure 5: XRD analyze of boehmite

Table 1: Independent variables for synthesized boehmite

Levels	Independent Variables		
	pH	Reaction temperature (ºC)	Reaction time (hour)
-1	4	170	16
0	8	185	20
+1	12	200	24

Table 2: Experimental design with Central Composite Design to optimize boehmite synthesized under specified operating condition

Run	Code Pattern			Variable (X_1, X_2, X_3)			Yield (%)	
				pH	Temp. (ºC)	Time (hour)	Experiment	Predicted
1	-1	-1	-1	4	170	16	95,16	95,13
2	-1	-1	+1	4	170	24	95,63	95,72
3	-1	+1	-1	4	200	16	95,57	95,64
4	-1	+1	+1	4	200	24	96,08	96,10
5	+1	-1	-1	12	170	16	96,07	96,10
6	+1	-1	+1	12	170	24	96,13	96,11
7	+1	+1	-1	12	200	16	97,14	97,10
8	+1	+1	+1	12	200	24	96,91	96,99
9	-α	0	0	1,2728	185	20	94,97	94,91
10	+ α	0	0	14,7272	185	20	96,48	96,47
11	0	- α	0	8	159,7731	20	96,43	96,41
12	0	+ α	0	8	210,2269	20	97,61	97,56
13	0	0	- α	8	185	13,2728	95,83	95,84
14	0	0	+ α	8	185	26,7272	96,31	96,23
15	0	0	0	8	185	20	98,23	98,31
16	0	0	0	8	185	20	98,38	98,31

Table 3: ANOVA for quadratic equation model in synthesized process

Source	Sum of Squares	df	Mean Square	F-value	p-value	
Model	14,97	9	1,66	217,99	<0,0001	Significant
X_1	2,95	1	2,95	386,97	<0,0001	
X_2	1,61	1	1,61	211,54	<0,0001	
X_3	0,1915	1	0,1915	25,11	0,0024	
X_1X_2	0,1225	1	0,1225	16,06	0,0071	
X_1X_3	0,1653	1	0,1653	21,67	0,0035	
X_2X_3	0,0078	1	0,0078	1,02	0,3506	
X_1^2	7,95	1	7,95	1042,72	<0,0001	
X_2^2	2,04	1	2,04	266,86	<0,0001	
X_3^2	6,0	1	6,0	786,27	<0,0001	
Residual	0,0458	6	0,0076			
Lack of Fit	0,0345	5	0,0069	0,6137	0,7421	Not Significant
Pure Error	0,0112	1	0,0112			
Total	15,01	15				
R^2	0,9964					
Adjusted R^2	0,9924					
Predicted R^2	0,9820					

Table 4: Critical values of the operating conditions of synthesized boehmite

Variable	Minimum	Critical	Maximum
pH	1,2728	9,0958	14,7272
Temperature (ºC)	159,7731	191,0421	210,2269
Time (Hour)	13,2728	20,1965	26,7272
Prediction of *Yield of Boehmite* : 98,45%			

XRD analysis is performed to determine the crystalline phase of the material. The crystallinity index and particle size can also be calculated by calculating the area of the diffraction peak shown. A sharp peak indicates the higher crystallinity of the synthesized product [22]. The results of the diffraction peaks detected in the XRD pattern show the presence of high peaks at 2θ angles of 13, 28, 39, 46, 49, 55, 65, and 72, which are characteristic of the diffraction peaks of boehmite [16]. The crystallinity index of the synthesized boehmite is 93.23%, this result is higher than the boehmite synthesized by Van Truong & Kim [2], which shows a crystallinity index of 69.11%. Particle size calculated by measuring the magnitude of the crystalline peak of the material, which will then calculated by the Scherrer equation. The particle size obtained by the Scherrer calculation is 24.5 nm. Research by Nemati Kharat et al., [4] showed the results of boehmite particle size in the range of 24.0-31.0 for processing at hydrothermal temperatures of 150-200ºC. Temperature affects the crystallinity of boehmite. In the ~200ºC range, the higher the hydrothermal temperature, the higher the crystallinity of boehmite. This indicated by the increasing sharpness of the diffraction intensity peak.

4. Conclusions

The synthesis of well-crystallized boehmite with the hydrothermal method was successful. The optimum operating conditions for the synthesis process are a pH of 9.1, a reaction temperature of 191ºC, and a reaction time of 20 hours and 12 minutes. The yield of the product treated at optimum operating conditions reached 98.49%. FTIR data shows that synthesized boehmite has a similar transmitant as commercial boehmite. XRD data shows that the crystallinity index reached 93, 23% and the average particle size was 24, 5 nm.

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