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Research Article

Application of Response Surface Methodology (RSM) to Study Transesterification of Palm Oil in the Presence of Zeolite-A as Catalyst

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Abstract
In this research, the application of response surface methodology with central composite design (RSM-CCD) to optimize transesterification of palm oil in the presence of zeolite-A as catalyst was investigated. Zeolite-A was synthesized from rice husk silica (RHS) and food-grade aluminium foil using hydrothermal method and then characterized using XRD and SEM. The synthesized zeolite was then applied to an optimized transesterification reaction using response RSM with three factorial levels, for three variables including methanol to oil ratio, catalyst load, and reaction time. The experimental results indicate that the yield of 99% was achieved at optimum conditions of methanol to oil volume ratio of 6, catalyst load of 9.6%, and reaction time of 4.3 hours. The results of experiments and predicted results based on the RSM model are in agreement as shown by the p-value less than 0.05 at a confidence level of 95%.

Keywords: palm oil, transesterification, biodiesel, zeolite-A, RSM

1. INTRODUCTION

Awareness of the depletion of petroleum reserves as a traditional source of fossil fuels for centuries and the environmental impacts that arise from fossil fuel application is a driving factor for increasing the use of renewable energy sources. Of particular importance are biomass-derived fuels (biofuels), biodiesel is one of them that has been used as a commercial fuel although still in the form of a blend with fossil diesel at a certain proportion. Biodiesel is a practical term for the product of transesterification, which is a reaction of vegetable oil or animal fat with simple alcohol, primarily methanol. In accordance with the chemical composition of vegetable oil, which is composed of free fatty acids (FFAs) and triglycerides, biodiesel production involves esterification of FFAs and transesterification of triglycerides. However, since glycerides are the prominent components of vegetable oil, transesterification is the main issue in biodiesel production. Several examples of vegetable oils that have been reported in the literature are waste cooking oil [1][2], coconut oil [3], *Ricinus communis* oil [4], rubber seed oil [5][6], and soybean oil [7]. Concerning this reaction, biodiesel is also known as a mixture of fatty acid methyl esters (FAMEs).

Regardless of its existence which has reached commercial status, the price of biodiesel is still higher than that of petrochemical diesel, reflecting the need for further effort to reduce production costs. In this respect, one of the production components that has been emphasized is the development of heterogeneous catalysts to replace homogeneous catalysts that remain as the prime catalyst up to the present. In search of effective heterogeneous catalysts, various solid catalysts have been developed. Several heterogeneous catalysts in the form of oxides reported are CaO [2][8], MgO [3][9], and calcium methoxide [10][11]. In the form of composite, several examples reported in the literature are NiO/ZSM-5 [4], MgO-CaO/SiO_2 [12], MgO/Fe_2O_3-SiO_2 [13], MgO/zeolite-A [14], and CaO/SiO_2 [15]. Besides composites, other catalysts that have been used are synthetic zeolites, such as zeolite-A [16], zeolite-X [17], and zeolite-Y [18].

In addition to the type of catalyst, other variables acknowledged to influence transesterification reaction are alcohol to oil ratio, reaction time, and catalyst load. Considering the role of these reaction variables, optimization of the transesterification reaction is generally carried out by conducting

Table 1. Experimental ranges and levels of the independent variables.

Variables	Symbol	Unit	Levels		
			-1	0	+1
Catalyst loads	A	%	5	7.5	10
Methanol to oil ratio	B	-	3	4	6
Reaction time	C	hour	3	4	5

experiments where one variable is varied while the others are kept constant. From such experiments, the optimum value for the variable being tested will be obtained, and this value is then used to determine the optimum value for other variables. This method has the disadvantage that it does not show an interactive effect and the overall effect of the variables involved [19]. To overcome these deficiencies, the use of multi-variable statistical techniques is now increasingly explored; one of them is known as response surface methodology (RSM) [7][20]. RSM is a statistical technique that uses multiple regression and correlation analysis as a tool to assess the effect of two or more independent variables on the dependent variable, with the aid of analysis of variance (ANOVA) [6][10][13][21]. This statistical method has been applied for the optimization of transesterification of different vegetable oils such as papaya oil [22], *Kusum* oil [23], and low-grade cooking oil [24].

The present work was conducted to study the application of response surface methodology (RSM) to optimize the transesterification of palm oil in the presence of zeolite-A as a catalyst. Zeolite-A was synthesized from rice husk silica (RHS) and food-grade aluminium foil using a hydrothermal method. Before use, the zeolite was characterized using XRD and SEM. The synthesized zeolite was then applied to an optimized transesterification reaction using RSM with three factorial levels for three variables including methanol to oil ratio, catalyst load, and reaction time.

2. MATERIALS AND METHODS

2.1. Materials and Equipment

Reagent grade sodium hydroxide (NaOH), nitric acid (HNO_3), and methanol (CH_3OH) were purchased from Merck. Rice husk and palm oil were obtained from local sources in Bandar Lampung. The main equipment used was an oven (Model Memmert UN Universal 321), furnace (Thermolyne Muffle Thermolyne 1100), X-ray diffraction instrument (XRD, Xpert MPD), scanning electron microscope (SEM, ZEISS EVO MA 10), and gas chromatography-mass spectrophotometry unit (GC-MS, QP2010S SHIMADZU).

2.2. Methods

2.2.1. Extraction of Rice Husk Silica

Rice husk silica extraction was carried out following the previously reported procedure [17]. In brief, the process involves several steps including mixing 50 g of dry rice husk with 500 mL of 1.5% NaOH solution, boiling the mixture for 30 min, aging the mixture at room temperature for 24 h, filtering the mixture to collect the filtrate (silica sol), neutralization of the sol using 10% HNO_3 solution to convert the silica from sol to gel, aging of the gel at room temperature for 24 h followed by rinsing of the gel with warm distilled water and completed by oven drying of the gel for 8 h at 100 ºC.

2.2.2. Synthesis of Zeolite-A

Zeolite-A was synthesized following the procedures reported by previous work [25]. To commence the experiment, a solvent was prepared by dissolving 20 g of NaOH in 250 mL of distilled water. A portion of 150 mL of the solvent was used to dissolve 30 g of rice husk silica, and the rest to dissolve 13.5 g of food-grade aluminum foil. The two solutions were mixed and magnetically stirred

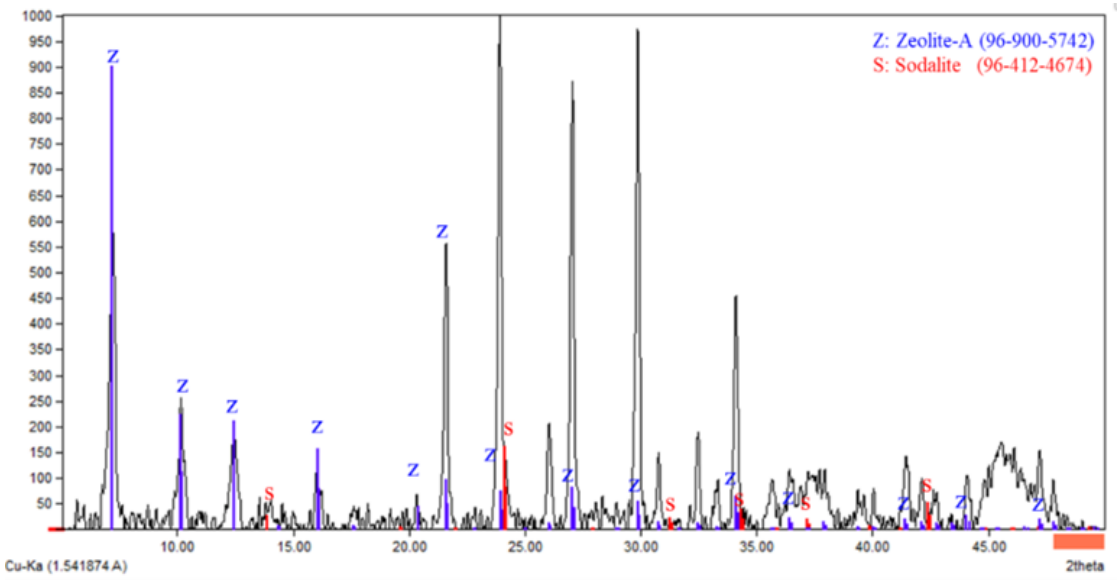

Figure 1. The diffractogram of synthesized zeolite-A.

for 3 h to homogenize the mixture, and then the mixture was transferred into an autoclave and allowed at room temperature for 24 h (aging process). After the completion of this aging process, the sample was subjected to crystallization by placing the autoclave in an oven at 100 °C for 72 h. The solid formed was then removed from the autoclave and then washed with distilled water until the rinsing water became neutral. The solid was oven-dried at 100 °C for 8 h, then calcined at 550 °C for 6 h, and finally ground into 250 mesh powder.

Table 2. Oil conversion from the experiments and predicted values.

Run	A: Catalyst load (%)	B: Methanol to oil ratio	C: Reaction time (h)	Oil conversion (%)	
				Actual	Predicted
1	5	3	5	34	31.19
2	10	6	5	100	98.76
3	7.5	4	5	52	55.75
4	7.5	3	4	48	53.96
5	7.5	4	5	60	55.75
6	5	6	4	48	47.64
7	10	3	5	48	52.02
8	7.5	4	3	44	45.99
9	10	4	3	72	65.11
10	7.5	4	5	60	55.75
11	7.5	3	4	60	53.96
12	7.5	6	3	56	54.62
13	10	3	5	56	52.02
14	5	3	4	40	37.47
15	5	4	5	40	38.27
16	10	4	3	60	65.11
17	5	4	5	32	38.27
18	10	6	4	99	101.98
19	7.5	3	5	40	44.23
20	5	3	3	20	21.17

Table 3. The results of data analysis using ANOVA.

Source	Sum of Squares	Degrees of Freedom	Mean Square	F-*value*	p-*value*	Remarks
Model	7130.88	9	792.32	24.75	< 0.0001	significant
A	3744.58	1	3744.58	116.99	< 0.0001	
B	1319.28	1	1319.28	41.22	< 0.0001	
C	323.70	1	323.70	10.11	0.0098	
AB	188.18	1	188.18	5.88	0.0358	
AC	33.46	1	33.46	1.05	0.3307	
BC	52.52	1	52.52	1.64	0.2291	
A^2	23.09	1	23.09	0.7213	0.4156	
B^2	0.1520	1	0.1520	0.0047	0.9464	
C^2	233.69	1	233.69	7.30	0.0222	
Residual	320.07	10	32.01			
Lack of Fit	69.40	4	17.35	0.4153	0.7928	not significant
Pure Error	250.67	6	41.78			
Total	7450.95	19				

$R^2 = 0.9570$ Predicted $R^2 = 0.8391$

2.2.3. Zeolite-A Characterization

The XRD diffractogram of the synthesized zeolite-A was generated by scanning the sample at (2θ) angle in the range of 0–50º on the instrument operated with Cu-Kα radiation (λ= 1.54 Å) with an energy of 40 kV and a current of 30 mA. The phase of the sample was identified with the aid of Match! Version 3.4.2 Build 96 software. The diffractogram of the sample was also compared with that of the standard zeolite-A provided in the International Zeolite Association (IZA) database. The surface morphology of the samples was examined using SEM, with an instrument operated at electron high tension (EHT) of 8 kV.

2.2.4. Transesterification Reaction

To experiment, an aliquot of 25 mL of oil was poured into a 250 mL round bottom flask and then a certain volume of methanol, to satisfy the predetermined methanol to oil ratio, and a predetermined mass of catalyst was added into the flask. A small magnetic stirring bar was added to the flask. The flask was immersed in a water bath paled on top of a hot plate with a magnetic stirrer

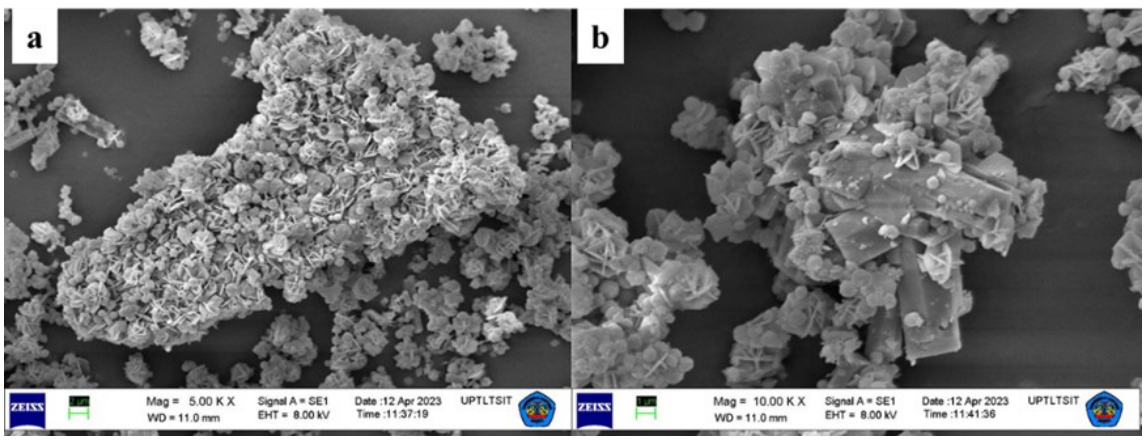

Figure 2. SEM micrograph of synthesized zeolite-A with the magnification of 5,000× (a) and 10,000× (b).

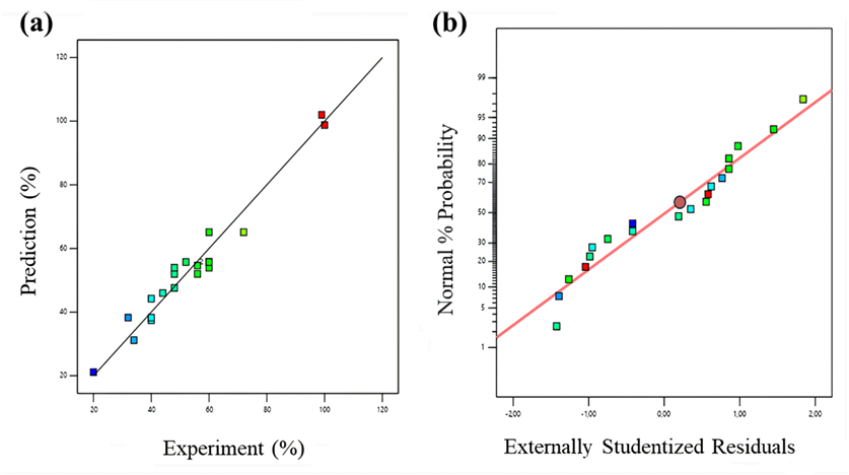

Figure 3. (a) Plot between experimental results and predicted results, and (b) plot of normal residue.

and connected with a reflux condenser with water as condensing liquid. The reaction was run at 70 ºC for a specified time. After the completion of the reaction, the sample was cooled to room temperature, and the product was filtered into a separatory funnel. The funnel was left overnight to allow separation between the biodiesel (upper layer) and residual oil (lower layer). The volume of the biodiesel was measured for the calculation of oil conversion using the following equation [5].

$$\%\ Conversion = \frac{V_i - V_f}{V_i}\ x\ 100\% \qquad (1)$$

In Equation 1, V_i = initial volume of oil and V_f = volume of unreacted oil. Identification of the fatty acid methyl esters composing the biodiesel was conducted using the GC-MS technique, with the aid of MS-Library software, NIST 62 and the Wiley 7 database.

2.2.5. Experimental Design

Optimization of the transesterification reaction was developed by application of response surface methodology with central composite design (RSM-CCD) using design expert 13.0 software. In this study, the following second-order polynomial model was proposed to predict response (Y) as a function of independent variables (Xi) and their interactions in Equation (2);

$$Y = \beta_0 + \sum_{i=1}^{k} \beta_i X_i + \sum_{i=1}^{k} \beta_{ii} X_i^2 + \sum_{i=1}^{k} \sum_{j=1}^{k} \beta_{ij} X_i X_j + \varepsilon \qquad (2)$$

where Y is a response, β_0, β_i, β_{ii}, and β_{ij}, indicate the regression coefficients for intercept effects, linear effects, quadratic effects, and double interactions, respectively. The model reliability of the model was evaluated in terms of the correlation coefficient, R^2 obtained from the analysis of variance (ANOVA). In this study, Y refers to biodiesel yield, and independent variables consist of catalyst load, methanol to oil ratio, and reaction time. The design of experiment (DOE) with the experimental ranges and levels of the independent variables is shown in Table 1.

3. RESULTS AND DISCUSSIONS

3.1. Zeolite-A Characterization

Figure 1 displays the XRD diffractogram and the phases composing the zeolite-A identified with the

Table 4. Criteria for optimization of reaction.

Variables	Goal	Lower limit	Upper limit
Catalyst load (%)	In range	5	10
Methanol to oil ratio	In range	3	6
Reaction time (h)	In range	3	5
Biodiesel conversion (%)	Maximum	20	100

Table 5. The results of model validation at optimum condition.

A: Catalyst load (%)	B: Methanol to oil ratio	C: Reaction time (h)	Prediction (%)	Experiment (%)	Error (%)
9.6	6	4.3	100	99	1

aid of Match! Version 3.4.2 Build 96 Software. As can be seen in Figure 1, two phases were identified, with zeolite-A as the main phase and sodalite as the second phase. In addition, the diffractogram also displays the existence of an amorphous phase as indicated by the presence of peaks with low intensity.

The existence of the sample as a multiphasic material is also indicated by the SEM micrograph in Figure 2.

As can be seen, the morphology of the sample is characterized by the existence of cubic-shaped particles, representing zeolite-A phase, spherical-shaped particles coated with rod-like structure, representing sodalite, and irregular shape which represents amorphous material.

3.2. Results of Transesterification

Based on DOE that has been previously determined (Table 1), 20 experiments were conducted and the percentages of conversion of the palm oil obtained from the experiments (actual result) were compared to the predicted conversion produced with the application of statistical software (design expert 13.0 software), as shown in Table 2. The data obtained from the experiments were then statistically analyzed using the ANOVA test to evaluate the effect of variables and the interaction between variables applied in the experiments. The results are compiled in Table 3. As can be seen in Table 3, the p-values of less than 0.05 at the 95% confidence level are obtained for the variables A, B, C, AB, and C^2, suggesting that these variables are significant factors in determining the yield of the reaction. For the variables AC, BC, A^2, and B^2 the opposite is true. The suitability of the model was also evaluated based on several other criteria, including insignificant lack of fit, a coefficient of determination (R^2) that is close to 1, the difference between adjusted R^2 and predicted R^2 is less than 2, and adequate precision is more than 4.

Based on the results of ANOVA as shown in

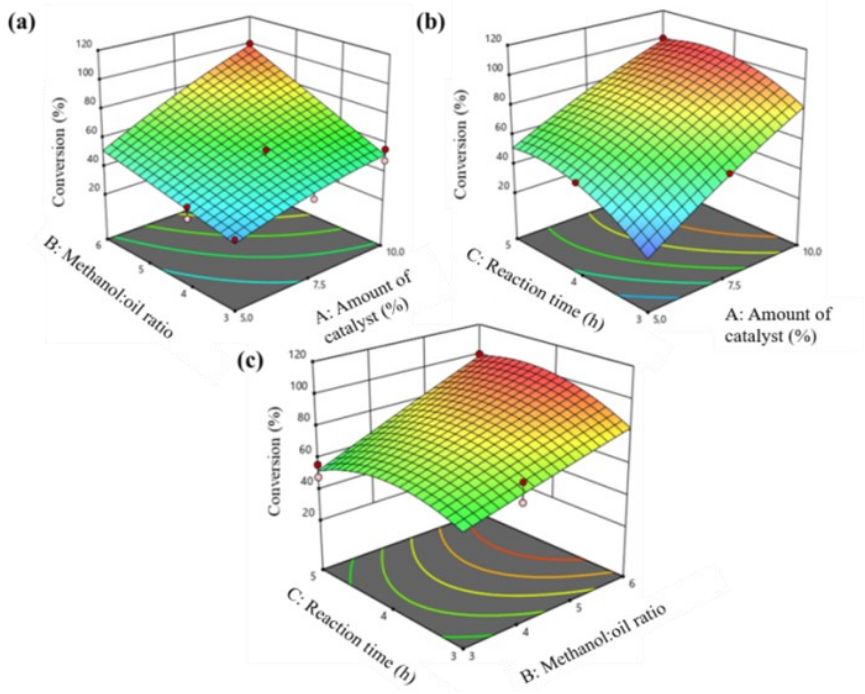

Figure 4. The 3D plot showing the effect of interaction between catalyst load and methanol to oil ratio (a), interaction between catalyst load and reaction time (b), and interaction between methanol to oil ratio and reaction time (c).

Table 3, the following model equation was suggested:

$$Y = 66{,}11 + 20{,}52A + 11{,}74B + 6{,}54C + 6{,}65AB - 3{,}45AC + 4{,}98BC - 2{,}63A^2 + 0{,}4160B^2 - 11{,}29C^2 \quad (3)$$

The above model is a second-order polynomial quadratic equation, with Y representing biodiesel yield, A, B, and C representing catalyst load, methanol to oil ratio, and reaction time, respectively. The positive sign indicates that the factor enhances the conversion of the oil into biodiesel, while for a negative sign, the opposite is true [26]. The suitability of the model is also supported by the plot between experimental and predicted results as well as the residual normal plot as presented in Figure 3. As displayed in Figure 3 (a), the plot produces a linear line, indicating that the predicted values are very close to the experimental values. Figure 3 (b) shows that the residual points are distributed around the diagonal line ideally, indicating that the residuals are normally distributed [27].

The 3D plot presenting the effect of the interaction of the variables applied in transesterification (reaction time, ratio of methanol to oil, and catalyst load) on biodiesel yield can be seen in Figure 4. Overall, the plots in Figure 4 display the effect of variables and their interaction on oil conversion, with the particular purpose of obtaining the value of each of the variables which leads to the optimum conversion of the oil treated.

The effect of the interaction between the catalyst load and the methanol to oil ratio at a constant time of 5 h is shown in Figure 4 (a). The 3D plot shows that the increase in the methanol to oil ratio and the amount of catalyst is in line with the increase in biodiesel yield. Figure 4 (b) shows a 3D plot for the interaction effect between the amount of catalyst load and reaction time on biodiesel yield, at a constant methanol to oil ratio of 6:1. The response surface shows that the yield of biodiesel increases with increasing loads of catalyst used. Figure 4 (c) is a 3D plot for the interaction effect between the methanol to oil ratio and reaction time at a constant catalyst load of 10%, showing that the yield of biodiesel increases with increasing methanol to oil ratio.

In Figure 4 (b) and (c) it can be observed that there is a curve associated with the reaction time, which indicates that increasing reaction time causes an increase in biodiesel conversion to the optimum point, followed by a decrease when the optimum point was exceeded. In this respect, it can be implied that reaction time only slightly influences the biodiesel yield. This also means that the interaction between the catalyst load and methanol to oil ratio is not significant in increasing biodiesel yield. This is in accordance with the ANOVA results (Table 3) which show that the interaction between catalyst load and reaction time as well as the interaction between reaction time and methanol to oil ratio are not significant in influencing biodiesel formation, as indicated by a p-value higher than 0.05. The optimization criteria for the transesterification reaction applied in this study are presented in Table 4.

Table 5 shows the optimum conditions with predicted and validated conversion results, with a relatively small error percentage of 1%. This shows that the proposed statistical model is suitable for optimizing the process of transesterification of palm oil with zeolite-A catalyst.

Many workers have reported the application of different zeolite-based catalysts for biodiesel production from different feedstocks. Several examples are presented in Table 6. As can be seen in Table 6, the conversions achieved are varied

Table 6. Zeolite-based catalysts applied for biodiesel production.

Catalyst	Feedstock	Optimum conversion (%)	Ref.
ZnO-β-zeolite	Waste cooking oil	84.10	[28]
Zeolite-Y	Waste cooking oil	84.44	[18]
ZSM-5	*Ricinus communis* oil	96.00	[5]
Zeolite- β	Waste sunflower oil	66.30	[29]

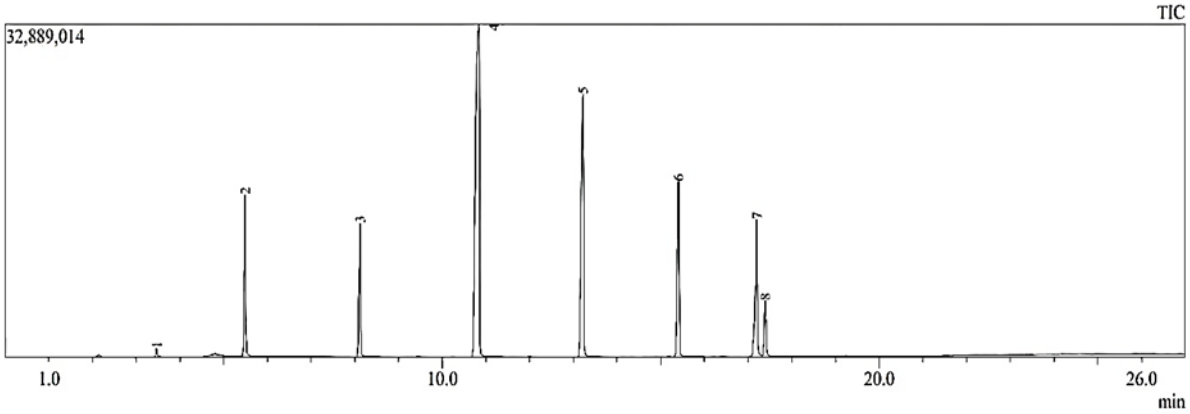

Figure 5. GC-chromatogram of biodiesel produced from transesterification of palm oil.

depending on the types of catalyst and feedstock treated. Compared to the examples presented in Table 6 the conversion achieved in this study is relatively higher.

A typical example of GC chromatogram of the biodiesel produced is shown in Figure 5. The chromatogram is characterized by the presence of 8 well-separated peaks with varied intensities. This feature suggests that there are 8 compounds composing the sample.

The components were then identified with the aid of mass spectrometry database (WILEY7.LIB), and the results are presented in Table 7. As can be seen in Table 7, the eight components of the sample are methyl esters that correspond to the fatty acid composition of palm oil reported in the literature. For example, the composition of palm oil reported in the literatures is presented in Table 8.

As can be seen in Table 8, some composition variations between the palm oils from different regions are observed. Regardless of such variations, it can be seen that palmitic and oleic acids are the main components of the oils.

4. CONCLUSIONS

In this study, zeolite-A was successfully synthesized as confirmed by the results of characterization using XRD and SEM. The synthesized zeolite-A was found to have high activity as a catalyst for the transesterification of palm oil into biodiesel, as demonstrated by the results of GC-MS analysis which confirms the presence of fatty acid methyl esters (FAMEs) as components of the biodiesel produced. Application response surface methodology with central composite design (RSM-CCD) to study the effect of three reaction variables (methanol to oil ratios, catalyst loads, and reaction times) revealed that the variables and their interactions are influencing factors for transesterification studied, with the statistical model of $Y = 66,11 + 20,52A + 11,74B + 6,54C + 6,65AB - 3,45AC + 4,98BC - 2,63A^2 + 0,4160B^2 - 11,29C^2$. Based on the 3D plots

Table 7. Chemical composition of biodiesel produced from transesterification of palm oil.

Peak no	Retention time (min)	Component name	Molecular formula	Relative amount (%)
1	3.461	Methyl caproate	$C_7H_{14}O_2$	0.40
2	5.488	Methyl caprylate	$C_9H_{18}O_2$	7.76
3	8.117	Methyl caprate	$C_{11}H_{22}O_2$	6.91
4	10.219	Methyl laurate	$C_{13}H_{26}O_2$	41.37
5	13.405	Methyl myristate	$C_{15}H_{30}O_2$	20.18
6	15.405	Methyl palmitate	$C_{17}H_{34}O_2$	10.75
7	17.195	Methyl oleate	$C_{19}H_{36}O_2$	9.60
8	17.391	Methyl stearate	$C_{19}H_{38}O_2$	3.04

Table 8. Fatty acid components of palm oil reported in the literatures.

Fatty acid	Relative quantity (%)		
	[30]	[31]	[32]
Caproic acid	nd	nd	nd
Caprylic acid	nd	0.2	nd
Capric acid	nd	0.3	nd
Lauric acid	0.1	2.4	0.2
Myristic acid	1.0	1.6	1.6
Palmitic acid	44.0	49.1	39.8
Palmitoleic acid	nd	0.3	0.2
Oleic acid	41.2	37.4	42.5
Stearic acid	5.0	4.5	4.4
Linolenic acid	0.5	9.7	0.4
Linoleic acid	8.0	0.5	11.2
Arachidonate acid	nd	nd	0.4

generated, it was found that the optimum conditions for transesterification of palm oil in the presence of zeolite-A as catalyst consists of a catalyst load of 9.6%, methanol to oil ratio of 6:1, and reaction time of 4.3 h to achieve the predicted oil conversion of 100%. From the experiment, the optimum oil conversion of 99% was achieved, indicating that the experimental design is in agreement with the generated statistical model.

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Conflicts of Interest

The author(s) declare no conflict of interest.

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