

Biodiesel Production from Vegetable Oil over Plasma Reactor: Optimization of Biodiesel Yield using Response Surface Methodology

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Abstract

Biodiesel production has received considerable attention in the recent past as a renewable fuel. The production of biodiesel by conventional transesterification process employs alkali or acid catalyst and has been industrially accepted for its high conversion and reaction rates. However for alkali catalyst, there may be risk of free acid or water contamination and soap formation is likely to take place which makes the separation process difficult. Although yield is high, the acids, being corrosive, may cause damage to the equipment and the reaction rate was also observed to be low. This research focuses on empirical modeling and optimization for the biodiesel production over plasma reactor. The plasma reactor technology is more promising than the conventional catalytic processes due to the reducing reaction time and easy in product separation. © 2009 BCREC. All rights reserved.

Keywords: biodiesel; plasma reactor; optimization; response surface methodology; central composite design

1. Introduction

Biodiesel has gained importance in the recent past year as a renewable fuel. The environmental issues concerned with the exhaust gases emission by the usage of fossil fuels also encourage the usage of biodiesel which has proved to be eco-friendly far more than fossil fuels. Biodiesel is a mixture of mono-alkyl esters obtained from vegetable oils like soyabean oil, jatropha oil, rapeseed oil, palm oil, sunflower oil, corn oil, peanut oil, canola oil and cottonseed oil. The direct usage of vegetable oils as biodiesel is possible by blending it with conventional diesel fuels in a suitable ratio. But direct usage of these triglyceric esters (oils) is unsatisfactory and impractical for long term usages in the

available diesel engines due to high viscosity, acid contamination, free fatty acid formation resulting in gum formation by oxidation and polymerization and carbon deposition. Hence vegetables oils are processed so as to acquire properties (viscosity and volatility) similar to that of fossil fuels and the processed fuel can be directly used in the diesel engines available. Three processing techniques are mainly used to convert vegetable oils to fuels form, they are pyrolysis, microemulsification and transesterification [1-4].

The most popular method of producing biodiesel is the transesterification of vegetable oils [1-4]. Biodiesel obtained by transesterification process

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is a mixture of mono-alkyl esters of higher fatty acids. Transesterification is the alcoholysis of triglyceric esters resulting in a mixture of mono-alkyl esters and glycerol. The transesterification process can be done in a number of ways such as using an alkali catalyst, acid catalyst, biocatalyst, heterogeneous catalyst or using alcohols in their supercritical state. In the alkali process, sodium hydroxide (NaOH) or potassium hydroxide (KOH) is used as a catalyst along with methanol or ethanol. This process is the most efficient and least corrosive of all the processes and the reaction rate is reasonably high even at a low temperature of 60 °C. There may be risk of free acid or water contamination and soap formation is likely to take place which makes the separation process difficult [1,2,5]. The second conventional way of producing biodiesel is using an acid catalyst instead of a base. Any mineral acid can be used to catalyze the process; the most commonly used acids are sulfuric acid and sulfonic acid. Although yield is high, the acids, being corrosive, may cause damage to the equipment and the reaction rate was also observed to be low [2].

A few published works on laboratory production and testing of biodiesel productions as alternative fuel have produced promising results [1-5]. Optimization of process parameters has also been conducted by previous researchers but focuses on the conventional transesterification process [6]. However until now, production of biodiesel from vegetable oil using plasma reactor technology has not been explored in the literatures. The plasma reactor technology is expected to reduce the reaction time considerably due to high energetic electrons role [7-8] and makes the separation processes to be easy due to no glycerol formation during the processes. The high energetic electrons were expected to replace the conventional chemical catalyst during the transesterification reactions. The review on design and principles of plasma reactor as a chemical reactor is explored in literature [7].

The purpose of this paper is to study the optimization of process parameters in biodiesel production over plasma reactor which maximizes the biodiesel product yield and minimizes the acid number of biodiesel. The optimization was done using response surface methodology (RSM). In the RSM method, a Central Composite Design (CCD) was used to design the experimental design to vary the independent variables. The adequacy of the proposed model was checked with the analysis of variance (ANOVA) using Fisher *F*-test.

2. Materials and Method

2.1. Materials and Plasma Reactor Configuration

The materials used in this research are: palm oil (Sania brand), Methanol (MERCK), KOH (MERCK), and ethanol (MERCK). The transesterification reaction is conducted in a plasma reactor. The plasma reactor consists of a DC High Voltage (HV) source and a Plasma Reactor (corona type). In the high voltage source, the AC source regulator was used to control the input voltage. The voltage applied to the reactor was varied according to the design of experiment within the range of 400 to 1200 kV which is dependent of the HV source capability. The reactor installation was designed for batch and continuous processes.

The palm oil and the methanol with the certain volume ratio (oil/methanol ratio) were flowed to a mixing tank. The mixed reactants were stirred and heated up to temperature of 65-70 °C. The hot mixed fluid was then flowed to the plasma reactor. The high voltage generator was then charged to the plasma reactor through a high voltage electrode and a ground electrode for certain charging time which is dependent of the design of experiment. After reaction was finished, the product was flowed to the settler (cone settler) to separate the unreacted methanol from biodiesel product. The biodiesel product was then characterized its properties, i.e. density (ASTM D 1298), viscosity (ASTM D445), acid number (ASTM D 664), and chemical composition (GC-MS). The GC-MS (SHIMADZU QP2010S equipped with RTX-5 MS column) analysis was applied to identify the chemical composition of biodiesel product.

2.2. Experimental Design and Method of Analysis

2.2.1. Central Composite Orthogonal Design

A central composite orthogonal design (CCOD) for three factors was employed for experimental design [9]. The value of α for orthogonality depends on the number of points in the factorial portion of the design which is given in Equation (1) [9-10].

$$\alpha = \left(\left[(n_c + n_s + n_o)^{1/2} - n_c^{1/2} \right]^2 \cdot n_c / 4 \right)^{1/4} \quad (1)$$

where n_c is the number of cube points in the design, n_s is the number of star points, and n_o is the number of center points. In this experimental de-

Table 1: Range and levels of independent variables for biodiesel yield optimization

Independent Variables	Range and levels of independent variables				
	Star point (-α)	Low level (-1)	Center point (0)	High level (+1)	Star point (+α)
Voltage (volt)	396	500	750	1000	1104
Volume Ratio Oil/methanol	0.22	0.25	0.38	0.50	0.55
Charging time (minutes)	2.9	5	10	15	17.1

sign, the center point was repeated four times so that there are 18 experimental runs (8 cube points, 6 star points, and 4 center points).

Three independent variables or factors, namely, voltage applied (volt), volume ratio of oil/methanol, and charging time (minutes), were selected as controlled factors. The ranges of independent variables were chosen based on the conditions from the variables screened prior to optimization. The ranges and levels used in the experimental work are given in Table 1 in which X_1 denotes voltage applied (volt), and X_2 denotes volume ratio of oil/methanol, and X_3 denotes charging time (minutes). In the experimental design, all variables are coded for statistical calculation according to Equation (2) below [9-10]:

$$x_i = \frac{\alpha [2X_i - (X_{\max} + X_{\min})]}{X_{\max} - X_{\min}} \quad (2)$$

where x_i is the dimensionless coded value of the i^{th} variable, X_i is the natural value of the i^{th} variable, X_{\max} and X_{\min} are the highest and the lowest limit of the i^{th} variable, respectively.

The responses and the corresponding factors are modeled and optimized using the response surface methodology (RSM) [9]. The RSM is a technique consisting of: (a) designing of experiments to provide adequate and reliable measurements of the response, (b) developing a mathematical model having the best fit to the data obtained from the experimental design, and (c) determining the optimal value of the independent variables that produces maximum or minimum value of the response. In this paper, the design of experiment and the response surface analysis were employed by utilizing STATISTICA version 6 software (StatSoft Inc., Tulsa, USA).

2.2.2. Model Fitting and Statistical Analysis

A quadratic polynomial equation was developed to predict the response as a function of independent variables and their interactions [9-10]. In general, the response for the quadratic polynomials is described in Equation (3). In this equation, Y is the predicted response, β_0 is the intercept coefficient, β_j are the linear terms, β_{jj} are the squared

$$Y = \beta_0 + \sum_{j=1}^3 \beta_j X_j + \sum_{j=1}^3 \beta_{jj} X_j^2 + \sum_{i < j} \beta_{ij} X_i X_j \quad (3)$$

terms, β_{ij} are the interaction terms, and X_i and X_j represent the uncoded of the independent variables. For each experimental factor the variance was partitioned into components, linear, quadratic and interaction, in order to assess the adequacy of the second order polynomial function and the relative importance or significance of the terms.

Three-dimensional response surfaces and contour plots were used for facilitating a straightforward examination of the influence of experimental variables on the responses. The coefficients of the models for the three responses were estimated with multiple regression analysis. The fit quality of the models was judged from their coefficients of correlation and determination. The adequacy of each model was checked with the analysis of variance (ANOVA) using Fisher F -test [9-10]. The significance of the equation parameters for each response was assessed by p -value and Student's t -test. The significance test of regression is attempted to determine the relationship between the response variable and a subset of the independent variables.

The steepest ascent method is used by the RSM to search for an optimal condition in which each response achieved a maximum value. The path of the steepest ascent is the direction such

Table 2: Experimental design matrix and results for biodiesel production using plasma reactor

Run No.	Independent Variables			Dependent Variables	
	Voltage (volt)	Volume Ratio of Oil/Methanol	Charging time (minutes)	Biodiesel Yield (%)	Acid Number (mg KOH/g)
1	500	0.25	5	23.24	0.724
2	500	0.25	15	18.36	0.724
3	500	0.50	5	36.52	0.845
4	500	0.50	15	35.72	0.362
5	1000	0.25	5	23.24	0.362
6	1000	0.25	15	23.37	0.483
7	1000	0.50	5	35.29	0.241
8	1000	0.50	15	37.79	0.362
9	396	0.38	10	32.83	0.362
10	1104	0.38	10	28.81	0.362
11	750	0.22	10	16.74	0.604
12	750	0.55	10	37.62	0.241
13	750	0.38	2.9	24.20	0.241
14	750	0.38	17.1	22.91	0.362
15	750	0.38	10	28.97	0.241
16	750	0.38	10	25.94	0.241
17	750	0.38	10	28.14	0.241
18	750	0.38	10	26.09	0.241

that the response value increases most rapidly. The independent variables are varied in the direction of increasing response until the response reaches its maximum. The increment or path of steepest ascent is directly proportional to the signs and magnitudes of the regression coefficients in the fitted model. From the models, it is possible to obtain the optimal independent variables that maximize a specified objective function.

3. Results and Discussion

3.1. Optimization of Biodiesel Product Yield

The experimental design matrix and the results of plasma reactor testing are presented in Table 2 consisting of 18 sets of coded conditions expressed in the natural values. The sequence of experiment was randomized in order to minimize the effects of uncontrolled factors. Objective of this section is to determine the optimal values of

applied voltage (X_1), volume ratio of oil/methanol (X_2), and cracking time ($-X_2$) such that a maximum biodiesel product yield is achieved. The three factors should be optimized simultaneously in order to include interaction effect among the variables. The experimental runs were based on the experimental design matrix shown in Table 2.

Coefficients of the empirical models built in Equation (3) are estimated using multiple regression analysis technique. The empirical mathematical model of the biodiesel product yield (Y_1) is presented in Equation (4).

The appropriateness of the models is judged from the determination coefficient, R^2 , which reveals a total variation of the observed values of activity about its mean [9-11]. In this fitting, the regression coefficients are estimated about 0.96. The R^2 value means a good agreement between the experimental and predicted values of the fitted model. It implies that 96 % of the total variation in the response is justified by the model. The correla-

$$Y_1 = 0.00004 X_1^2 - 0.06133 X_1 + 54.18083 X_2^2 + 17.57605 X_2 - 0.04530 X_3^2 - 0.28353 X_3 - 0.01759 X_1 X_2 + 0.00083 X_1 X_3 + 1.28774 X_2 X_3 + 37.15589 \quad (4)$$

Table 3: ANOVA study on the biodiesel product yield model

Factor	SS	df	MS	F-value
SS Regression	708.2721	9	78.6969	23.4901
SS Error	28.0438	8	3.3502	
SS total	733.2000	17		
R ²			0.96	

Note: SS : Sum of Square; df : degree of freedom; MS : mean square; and $F_{table(0.05, 9, 8)} = 3.39$

Table 4: Regression coefficients in the biodiesel yield empirical model and their significance effect

Parameters	Terms	Coefficient	t-value	p-value
β_0	Intercept	37.15589	3.14295	0.013744
β_1	X_1	-0.06133	-3.28785	0.011057
β_1^2	X_1^2	0.00004	3.76362	0.005516
β_2	X_2	17.57605	0.46696	0.652983
β_2^2	X_2^2	54.18083	1.26026	0.243092
β_3	X_3	-0.28353	-0.36426	0.725103
β_3^2	X_3^2	-0.04530	-1.72002	0.123742
$\beta_1\beta_2$	X_1X_2	-0.01759	-0.83061	0.430282
$\beta_1\beta_3$	X_1X_3	0.00083	1.56922	0.155237
$\beta_2\beta_3$	X_2X_3	1.28774	1.21618	0.258580
R ²			0.96	

tion between the experimental and the predicted values is also acceptable

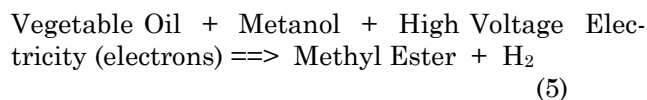
The adequacy of the model was checked with the analysis of variance (ANOVA) as presented in Table 3. The calculated F -value should be greater than the tabulated value as the model to be considered good fitting. In fact, the F -value corresponding to the biodiesel product yield response is 23.49 and exceeds the tabulated F -value ($F_{0.05,9,8} = 3.39$) as presented in Table 3. The F -value shows a statistically significant regression at 95 % confidence level. In this case, the null hypothesis (H_0) is rejected at 5 % level of significance based on the marked F -value [9-10] implies that at least one of the independent variables contributes significantly to the model. Comparison between the predicted and the observed biodiesel product yield is presented in Figure 1. From the figure, it is shown that the the predicted value of biodiesel product yield closes to the observed values.

The results in Table 4 explain the multiple regression results and the significance of the model

regression coefficients with respect to the biodiesel product yield. The p - and Student's t -values are used to check the significance of each term at a specified level of significance [9]. The p -value is defined as the smallest level of significance that would reject the null hypothesis, H_0 . The smaller the magnitude of the p -value the more significant is the corresponding coefficient and contributes largely towards the response variable. From the table, the quadratic of applied voltage term (X_{12}) has the largest effect on the biodiesel product yield at the 99 % confidence level as indicated by the lowest p -value (<0.01) and the highest absolute t -value (3.764). Next, the linear term of applied voltage (X_1) shows a substantial significant effect at 95 % confidence level (p -value < 0.05) followed by the effect of quadratic charging time (X_{32}) and interaction between charging time and applied voltage term (X_1X_3). From the p - and t - values studies, it can be implied that the empirical model can be used to simulate the biodiesel product yield as function of the three process parameters.

$$Y_1 = 0.000001 X_1^2 - 0.00329 X_1 + 7.7446 X_2^2 - 5.57106 X_2 + 0.00235 X_3^2 - 0.06666 X_3 - 0.00006 X_1 X_2 + 0.00007 X_1 X_3 - 0.09499 X_2 X_3 + 3.11785 \quad (6)$$

Three-dimensional surface plot of the biodiesel product yield model shown in Figures 2 and 3 represent estimated response over the process parameters (applied voltage, volume ratio of oil/methanol, and charging time). Increasing the voltage applied to electrode of the plasma reactor leads to higher biodiesel product yield. The higher the applied voltage, the higher the electrons flows from the high voltage electrode to the ground electrode. The higher the energetic electrons lead to stronger the cracking and the recombination of chemical bonds during the reaction as represented in Equation (5) below:



The reaction as written in Equation (5) is suggested from the fact that there is no soap formation during the reaction. The gas was produced during the transesterification reaction over the plasma reactor.

The applied voltage gives more significant effect than the charging time and volume ratio of reactants. This fact implies that the energetic electrons have dominant role during the reaction. However, the reaction should be controlled by regulating their power in order to prevent exces-

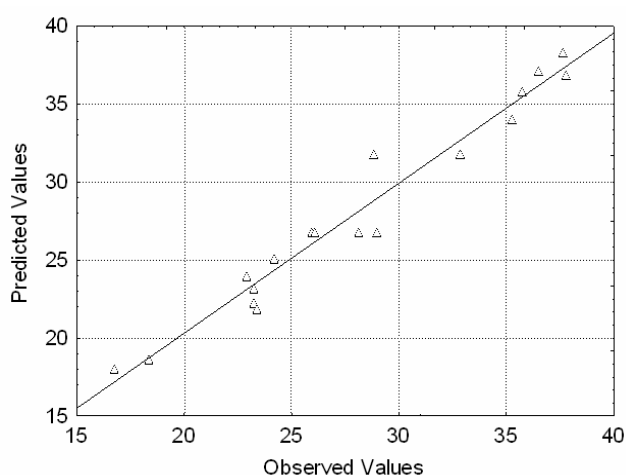


Figure 1: Comparison of predicted and observed values of biodiesel product yield

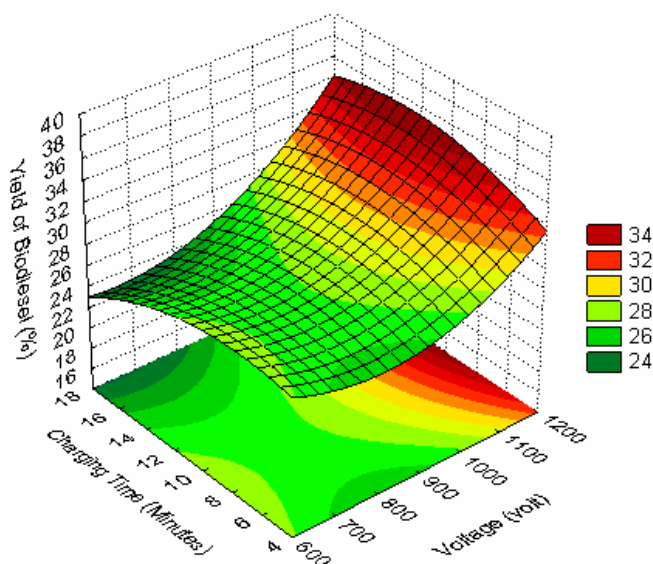


Figure 2: Effect of applied voltage and charging time on the biodiesel product yield

Table 5: ANOVA study on the empirical model of the acid number of biodiesel product

Factors	SS	df	MS	F-value
SS Regression	0.5040	9	0.0560	3.42
SS error	0.1312	8	0.0164	
SS total	0.6420	17		
R ²		0.81		

Note: SS : Sum of Square; df : degree of freedom; MS : mean square; $F_{table(0.05, 9, 8)} = 3.39$

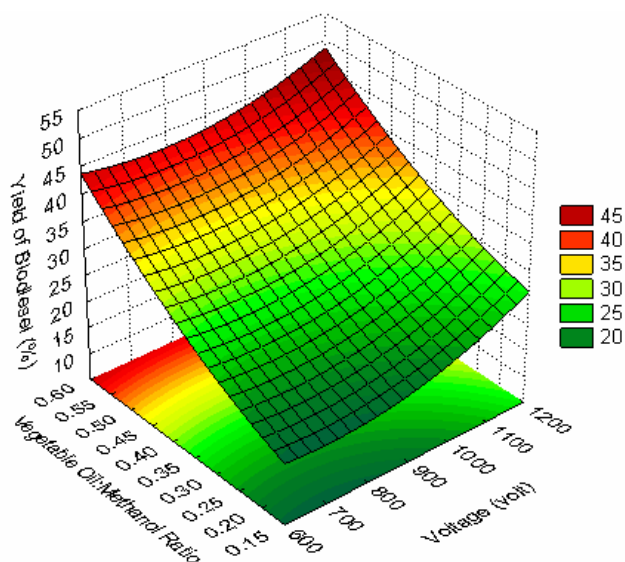


Figure 3: Effect of applied voltage and volume ratio of oil/methanol on the biodiesel product yield

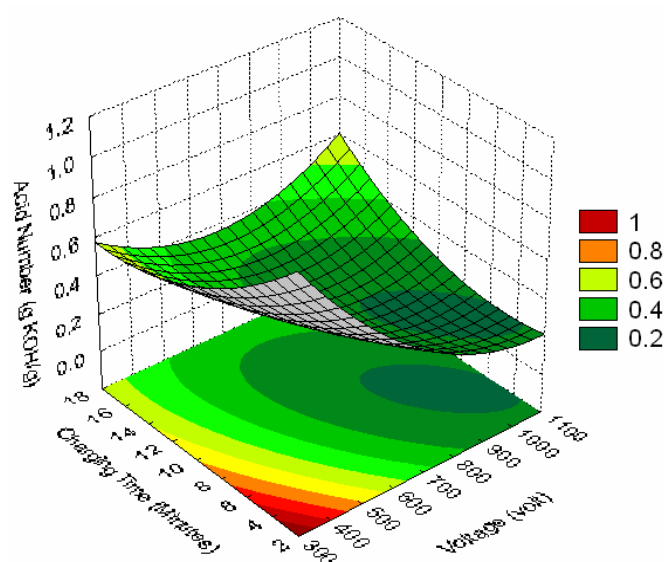


Figure 4: Effect of applied voltage and charging time on the acid number of biodiesel product

sive cracking of chemical bond in methyl esters. The charging time should be controlled to be as short as possible, particularly at higher applied voltage. The shorter charging time could reduce the reaction time. The shorter reaction time is expected in order to be more efficient process than the conventional transesterification process (more than 60 minutes) [1-5]. The other advantages of plasma reactor technology for biodiesel production are easy in separation or purification process due to no soap and glycerol formations during the plasma reaction.

The optimum operating conditions are achieved at higher applied voltage (about 1100 volt), mild charging time (about 11 minutes), and certain oil/methanol ratio to obtain a maximum biodiesel product yield of about 35%. However, the oil/methanol ratio should be studied so far to get

the optimal conditions. The applied voltage ranges are suggested to be higher than 1 kV up to 8 kV to achieve the higher energetic electrons. The applied voltage should be correlated with charging time parameters to achieve an efficient process

3.2. Minimization of Acid Number of Biodiesel Product

The experimental design matrix and the results of plasma reactor testing are presented in Table 2. Objective of this section is to determine the optimum values of applied voltage (X_1), volume ratio of oil/methanol (X_2), and cracking time ($-X_2$) such that a minimum acid number of biodiesel product is achieved. The three factors should be

Table 6: GC-MS analysis results for biodiesel product characterization

Retention time (minutes)	Chemical identification in the biodiesel product	Chemical Compositions (wt%)
21.917	Methyl palmitate ($C_{17}H_{34}O_2$)	19.46
23.008	Palmitic Acid ($C_{16}H_{32}O_2$)	19.73
25.417	Methyl Oleate ($C_{19}H_{36}O_2$)	17.45
26.450	Oleic Acid ($C_{18}H_{34}O_2$)	12.39
33.275	Diethyl phthalate ($C_{24}H_{38}O_4$)	21.67
37.658	Squalene ($C_{30}H_{50}$)	9.29

optimized simultaneously in order to include interaction effect among the variables.

Coefficients of the empirical models are estimated using multiple regression analysis technique. The empirical mathematical model of the acid number of biodiesel product (Y_2) is presented in Equation (6). In this fitting, the regression coefficients (R^2) are estimated about 0.81. It implies that 81 % of the total variation in the response is justified by the model. The correlation between the experimental and the predicted values is also acceptable

The adequacy of the model was checked with the analysis of variance (ANOVA) as presented in Table 5. The F -value corresponding to the acid number of biodiesel product is 3.42 and still exceeds the tabulated F -value ($F_{0.05,9,8} = 3.39$) as presented in Table 5. The F -value shows a statistically significant regression at 95 % confidence level. In this case, the null hypothesis (H_0) is rejected at 5 % level of significance based on the marked F -value [9-10] implies that at least one of the independent variables contributes significantly to the model.

Three-dimensional surface plot of the acid number of biodiesel product is shown in Figures 4 to 5 representing an estimated response over the process parameters (applied voltage, volume ratio of oil/methanol, and charging time). Increasing the voltage to the high voltage electrode of the plasma reactor leads to lower acid number of biodiesel product. However, charging time only affects slightly on the acid number. The lower the energetic electrons lead to lower the cracking power.

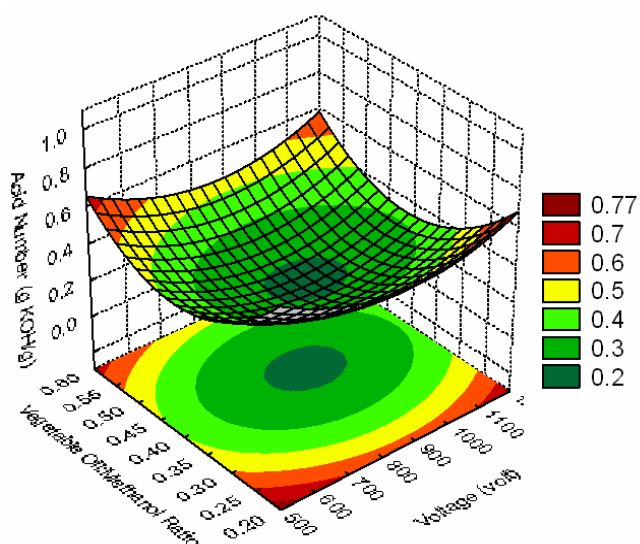


Figure 5: Effect of applied voltage and volume ratio of oil/methanol on the acid number of biodiesel product

Therefore, the recombination of chemical bonds to form methyl esters is not completely finished.

The applied voltage gives more significant effect than the charging time and volume ratio of reactants. This fact implies that the energetic electrons have dominant role during the reaction. However, the reaction should be controlled by regulating their power in order to prevent excessive cracking of chemical bond in methyl esters. The charging time should be controlled to be as short as possible, particularly at higher applied voltage. The shorter charging time could reduce the reaction time. The shorter reaction time is expected in order to be more efficient process than the conventional transesterification process (more than 60 minutes) [1-5].

The optimum operating conditions are achieved at higher applied voltage (about 948 volt), mild charging time (about 11 minutes), and oil/methanol ratio (0.4) to obtain a minimum acid number of biodiesel product of about 17%. However, the oil/methanol ratio should be studied so far to get the optimal conditions. The applied voltage ranges are suggested to be higher than 1 kV up to 8 kV to achieve the higher energetic electrons. The applied voltage should be correlated with charging time parameters to achieve an efficient process.

3.3. Biodiesel Product Characterizations

The hypothesis of methyl ester formation during the transesterification of vegetable oil and methanol over the plasma reactor is proven by chemical identification of biodiesel product using

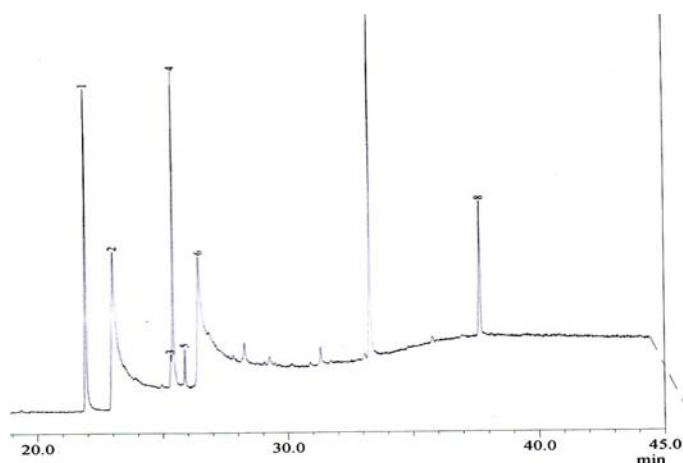


Figure 6: GC-MS analysis result of biodiesel product

GC-MS [12]. The GC-MS analysis result of the biodiesel product is presented in Figure 6 and Table 6. From the figure and the table, it is shown that the methyl ester (methyl palmitate, methyl oleate, and Dioctyl phthalate) were formed during the plasma reaction. This fact proofs the proposed reaction in Equation (5). Another important fact is that the interaction among applied voltage, charging time, and volume ratio of reactant is crucial for methyl ester formation. The energetic electrons have role as a catalyst giving a reaction path which having lower activation energy. The other characteristics of the biodiesel product through this technology are density of about 0.87 kg/m³ (ASTM D1298), acid number of about 0.40 mg KOH/g (ASTM D664), and viscosity of about 4.9 mm²/s (ASTM D445) [13-14]. However, the complete biodiesel product characterizations could not be listed here.

4. Conclusions

Biodiesel production from vegetable oil through plasma technology has been developed. The biodiesel product (methyl esters) can be produced without conventional chemical catalyst. The empirical models for prediction of biodiesel product yield and acid number over plasma reactor were produced. The optimum operating conditions are achieved at higher applied voltage (about 948 volt), mild charging time (about 11 minutes), and oil/methanol ratio (0.4) to obtain a minimum acid number of biodiesel product of about 17%. The applied voltage ranges are suggested to be higher than 1 kV up to 8 kV to achieve the higher energetic electrons. The applied voltage should be correlated with charging time parameters to achieve an efficient process. The plasma reactor technology is more promising than the conventional catalytic processes due to the reducing reaction time and easy in product separation. However, this research is still a preliminary research which should be more explored intensively .

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