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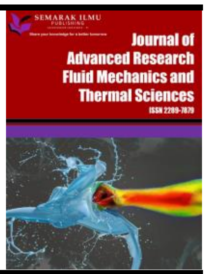

Optimization of Biodiesel Production from Beef Tallow Using Microwave Assisted

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ABSTRACT

The need for fuel in Indonesia is increasing every year. Renewable and environmentally friendly alternative fuels are the right choice to meet national energy need and can substitute fossil fuels. Biodiesel has been widely applied in diesel engines that run on diesel fuel. Biodiesel is processed from plant and animal oils or fats. Non-edible oil is preferable to be processed into biodiesel because it doesn't interfere with food availability. Beef tallow has abundant availability, the users are still few, and has an ALB content of <1%, making it suitable as a raw material for biodiesel. The transesterification reaction to process oil (triglycerides) into biodiesel (methyl ester) requires effective heating, one of which is the microwave method. Optimal process conditions are required to produce high biodiesel yields. Therefore, the data optimization process is carried out in order to obtain the optimum conditions and save the number of running. In this study using the reaction temperature and catalyst concentration (%weight) as independent variable. The fixed variables and were the mole ratio of methanol: oil (6:1), reaction time (20 minutes), power (200 watt) and stirring speed (200 rpm). Data analysis of optimization used the Response Surface Methodology (RSM) based on Central Composite Design (CCD) with the help of the software STASTICA 10. The research results the optimum yield was 98.736% at conditions of reaction temperature 74.142ºC and catalyst 0.396%weight. Mathematical equation for optimum conditions $y = 90.81073 - 8.74778\,X_1$.

1. Introduction

Indonesia is a country that has abundant natural resources, such as fossil fuels in the form of petroleum, coal, natural gas etc. Fossil fuels resources have been used as the main energy sources for a long time in Indonesia and other countries in the world. So that, the global needs and demand for fossil fuels tend to increase significantly every year. The data from British Petroleum shows years 2007 to 2018 the energy demand in Indonesia increase on the average 2.8% in 2007 to 2017 and in 2018 increased significantly by 4.9%. However, fuel production has declined 0.4% in 2007-2017 and in 2018 decreased up to 6.7% [1]. The decrease of production will result insufficient energy needs and affected to an energy crisis in the future. So, to resolve that issue the alternative fuels are needed

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for as substitute fossil fuels. As substitute fuels the alternative fuels has meet the criteria conditions, such as renewable resources, abundant availability, easy obtained, clean energy, and similar characteristics to fossil fuels.

Many research developments on alternative fuels have been carried out. The research is still needed on an ongoing basis to get results that are suitable and can be produced on a large scale. This continues to be done because it has proven its benefits, namely it is more environmentally friendly, biodegradable, and efficient. In addition, that us od alternative materials is needed to utilize natural materials that exist and have a low value to be converted into alternative fuels. Some of the alternative fuels that have been widely used are biodiesel, bioethanol, biogas [2]. Biodiesel is renewable, non-toxic and biodiesel has a higher flash point than diesel [3].

Biodiesel is a non-fossil based diesel fuel consisting of long chain mono-alkyl fatty acid esters processed from renewable lipid sources [4]. Biodiesel has been successfully applied as a renewable fuel as a mixture of diesel fuel which is often called biodiesel. So that, biodiesel has great potential as an alternative fuel that can replace diesel or diesel fuel.

The raw materials of biodiesel can be obtained from vegetable oils and animal fats [5]. That raw material is divided into two types, edible oil and non-edible oil, but it is preferred to use non-edible oil. This is because the edible is mainly used for food consumption, so it can disrupt the stock of food availability which will result a conflict between food and fuel needs. Because of that, using non-edible oil as the main source of biodiesel production is the best option [4]. In addition, the low price of non-edible oil raw materials and underused in the industry are driving factors in the processing of biodiesel from non-edible oils.

Recent studies have conducted several researches on biodiesel processing from low cost raw materials, including used cooking oil, new types of plant oils, and animal fats [6]. From some of these raw materials, research on biodiesel from animal fats is still a little done. So, more depth research is needed to study the potential of animal fat as a raw material for biodiesel.

The animal fats that can processed to biodiesel are beef tallow, chicken fat, lard, yellow fat, brown fat, and other weak animal fats [7]. Among these animal fats, tallow has high potential and several advantages over others. The advantage is that it is easy to obtain from slaughterhouses, is not maximally utilized due to excess residue from slaughtering animals into waste. So, tallow has more potential and advantages to be used as raw material for biodiesel [8]. Beef tallow has the highest ester content of 92.77% compared to pork, chicken, veal and goose [9].

Tallow is a non-edible oil fatty acid, low cost, and its use in industry is still limited [10]. Tallow is a lipid material composed of the main components of triglycerides and a small portion of monoglyceride [11]. In tallow, there are 45,6% saturated fatty acids. Saturated fatty acids have the advantage of being converted to methyl esters (biodiesel), which have a high cetane number and are not easily oxidized, but at high temperatures crystallization is easy. Tallow also has a high free fatty acid content, which is more than 8%, which is obtained from low-quality tallow called tallow waste [12]. High free fatty acids tend to form a saponification reaction so that it inhibits the transesterification reaction process which causes a low methyl ester yield. So, in the process of methyl ester production, pretreatment is needed to reduce levels of free fatty acids (FFA).

Animal fats with low FFA levels can be processed into biodiesel with a one-stage transesterification process using the help of an alkaline catalyst under low operating conditions [13]. This process reacts triglycerides with alcohol with an alkaline catalyst to form esters and glycerol. One of the base catalysts that are widely used in biodiesel synthesis is sodium hydroxide. This alkaline catalyst has the main advantage of being low in price. Transesterification reaction using alkaline catalyst faster rate, almost 4000 times faster than catalyzed with acid catalyst [14]. So that in this study used sodium hydroxide catalyst for the transesterification reaction.

In the process of synthesis biodiesel, an effective heating method is needed so that the required heat can be transferred optimally and is not wasted outside. The use of conventional heating methods requires a longer reaction time and a larger energy input because the transfer spreads out to be inefficient. The transesterification reaction using a microwave can save energy requirements and shorten the reaction time, because the energy transfer takes place molecularly [15]. Methanol as a reactant in the transesterification reaction which is a polar compound has a high microwave absorption capacity and is an organic solvent with high polarity. These two properties are factors that support the rapid transesterification reaction caused by dipolar polarization and ion conduction [16].

This research used Response Surface Methodology (RSM) for the optimization data to resulted maximum of yield biodiesel and optimum operation conditions. RSM is based on Central Composite Design (CCD) for the experimental design that can compute from STATISTICA 10 software.

This research studied the optimization of temperature and catalyst concentration in the process of producing biodiesel from tallow using a microwave. This research has great potential to continue to be developed and mass produced because it has several advantages. The main advantage is that it can produce biodiesel from low-cost raw materials and utilize tallow waste. The selection of tallow and sodium hydroxide catalyst is expected to reduce production costs to obtain biodiesel at a low price.

2. Methodology

2.1 Materials

The main ingredient used in this research is beef tallow from traditional markets in Wonogiri Regency, which is pre-treated by heating and filtering to get clean tallow oil. The chemicals used include analytical standard methanol with a level of 99.99%, pure sodium hydroxide in the form of pellets, pure KOH in the form of pellets, analytical standard ethanol, PP indicators, and analytical standard oxalic acid. The research tool used in this research is a series of microwave devices with modified temperature sets consisting of a microwave, a glass reactor, a thermometer, a magnetic stirrer, a spiral condenser, and a pump.

2.2 Methods

2.2.1 Pre-treatment

Solid beef fat is heated on the stove until the fat becomes a clear yellow liquid. beef oil is filtered to separate from the impurities. Liquid oil is stored in a closed container.

2.2.2 Transesterification assisted by microwave

The material was entered in the reactor with a molar ratio of oil and methanol 1: 6. The condition of beef oil was in the liquid phase. The microwave is turned on according to the desired operating conditions, after reaching a certain temperature the sodium hydroxide catalyst is introduced into the reactor. After reaching a certain time, the synthesis process is stopped. The product is removed from the microwave and separated between glycerol and biodiesel. Biodiesel is washed and then evaporated to remove water in the product. Biodiesel products were analyzed by GCMS to determine the concentration of methyl esters in biodiesel.

2.3 Experimental design

The research design uses CCD, there are two changing variables (k), namely the concentration of the catalyst with the code level X_1 and the reaction temperature with the code level X_2. In the CCD design, it is necessary to add an axial point, with a calculation of 2k, which is 4 axial points. And to get a rotatable CCD, it must have the right approach from the center point in the form of axial points around the center point, then it is calculated by the formula $(2k)^{1/4}$ = 1.414. The coding values for the variables X_1 and X_2 are presented in Table 1.

Table 1
Level code and experiment level value

Level Code	-1.414	-1	0	+1	+1.414
X_1	45.86	50	60	70	74.14
X_2	0.40	0.50	0.75	1.00	1.10

This study was designed with a response surface methodology (RSM) method based on central composite design (CCD) using software STATISTICA 10. The results of the experimental design are presented in Table 2.

Table 2
Experimental design

Run	X_1 Catalyst, %	X_2 Temperature, °C	Response Yield, %
1	-1	-1	
2	-1	+1	
3	+1	-1	
4	+1	+1	
5	-1.414	0	
6	+ 1.414	0	
7	0	-1.414	
8	0	+1.414	
9	0	0	
10	0	0	

2.3.1 Data analysis

Mathematical models that described the relationship and interaction between independent variables by following a second order polynomial equation as shown in Eq. (1) [17].

$$y = \beta_0 + \sum_{i=1}^{k} \beta_i x_i + \sum_{\substack{i=1 \\ i<j}}^{k-1} \sum_{j=2}^{k} \beta_{ij}\, x_i x_j + \sum_{i=1}^{k} \beta_{ii}\, x_i^2 + \varepsilon \quad (1)$$

where various x_i and x_j are independent variables, respectively, y is response variables; β_0, β_i, β_{ii}, β_{ij} are the regression coefficients and k is the number of variables.

3. Results

3.1 The Development Model for The Methyl Ester Yield Prediction

The research data are presented in Table 3. The research data shows that the yield of biodiesel produced ranges from 86% to 96% and the result shows that the reaction temperature and concentration of catalyst affect the biodiesel synthesis results obtained. Cunha [9] research that biodiesel production from beef tallow uses potassium hydroxide catalyst of 1.5%, ratio of methanol: oil of 6:1 and a reaction time of 180 minutes yield 95-97%. This research was conducted using a microwave with a reaction time of 20 minutes, the yield obtained has the same value as that of Cunha *et al.,* [18] with a reaction time of 180 minutes. Biodiesel production using microwave requires a shorter time than conventional methods.

Table 3
The set of experimental variables based on central composite design and observed response of methyl ester yield

Run	X_1 Catalyst, %	X_2 Temperature, ºC	Experiment value Yield, %	Predicted value Yield, %
1	0.50	50.00	95.10	93.74
2	0.50	70.00	96.31	96.51
3	1.00	50.00	87.19	84.65
4	1.00	70.00	89.08	88.09
5	0.40	60.00	96.15	96.48
6	1.10	60.00	82.11	84.11
7	0.75	45.86	86.73	89.00
8	0.75	74.14	93.33	93.39
9(C)	0.75	60.00	89.84	90.81
10(C)	0.75	60.00	91.79	90.81

Figure 1 show of the comparison of the prediction results with the results of the study is used to show the magnitude of the regression value between the two results. This figure contains a black circle that shows the comparison of the predicted value and experiment value and a red line that shows the regression line. The value of regression price (R^2) is 0.894. This value indicates that the results of the research data are close to the prediction data calculation, because the R^2 value is very close to the value of 1 or the maximum.

The mathematical equation at optimum conditions is the relationship between independent variables to get the value of the response at the optimum condition. This equation is compiled using the Effect Estimates coefficient data presented on Table 4, the data is analyzed and optimized using the first order polynomial equation. The significance value (p) is set below 0.05, if the p value <0.05, the parameter is considered to have a significant effect. And if the p value > 0.05, the parameter is considered not having a significant effect [19].

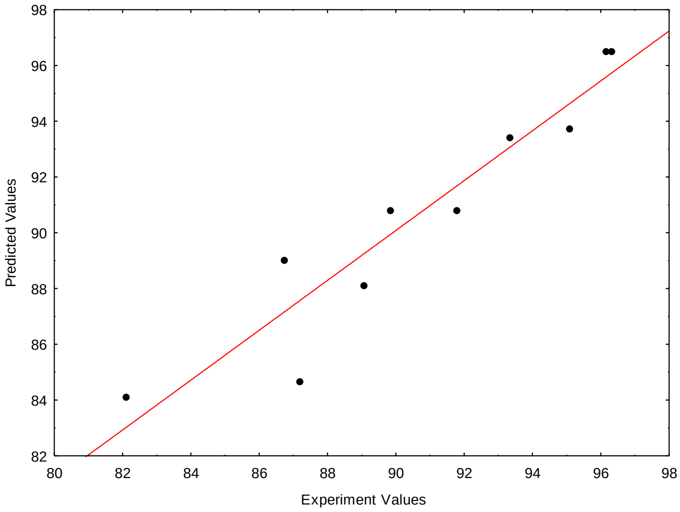

Fig. 1. The correlation between experimental value and prediction value

Table 4
Effect estimates coefficient and significance value

Factor	Effect	Std.Err.	T	p	
Mean/Interc.	90,81073	1,601628	56,69900	0.00000	
X_1	-8,74778	1,601628	-5,46180	0.00546	
X_1^2	-0,51010	2,118755	-0,24076	0.82158	p>0,05
X_2	3,11115	1,601628	1,94249	0.12402	p>0,05
X_2^2	0,39037	2,118755	0,18425	0.86278	p>0,05
X_1X_2	0,34100	2,265045	0,15055	0.88762	p>0,05
R^2	0,89414		Adj R^2	0.76182	

From the data analysis, a mathematical equation is obtained at the optimum conditions as follows

$$y = 90.81073 - 8.74778\, X_1 \tag{2}$$

In Figure 2 show the Pareto diagram, it can be seen that the effect value of the parameter that crosses the p = 0.05 line is a parameter that has a significant effect on yield, namely the concentration of catalyst (X_1). While the effect value of parameters that do not cross the p = 0.05 line is a parameter that does not have a significant and negligible effect, namely temperature (X_2), quadratic concentration of catalysts (X_{12}), quadratic temperature (X_{22}), interaction between variables of catalyst amount and temperature (X_1X_2) because the effect on yield is insignificant.

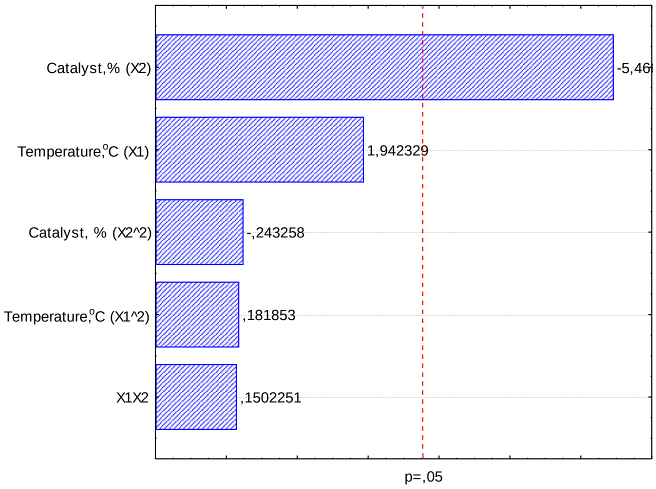

Fig. 2. Pareto Diagram

3.2 Optimization of Operating Conditions of Production Biodiesel from Beef Tallow

Surface Response Profile shows the relationship between independent variables of temperature and catalyst concentration on biodiesel yield. The surface response profile is presented in Figure 3. The figure shows that the higher the temperature and the lower the concentration of catalyst, the reaction time is constant (20 minutes) and the molar ratio of methanol:oil (6:1). The highest biodiesel yield was obtained at 96.31% and at a temperature of 70°C and a catalyst concentration of 0.5%.

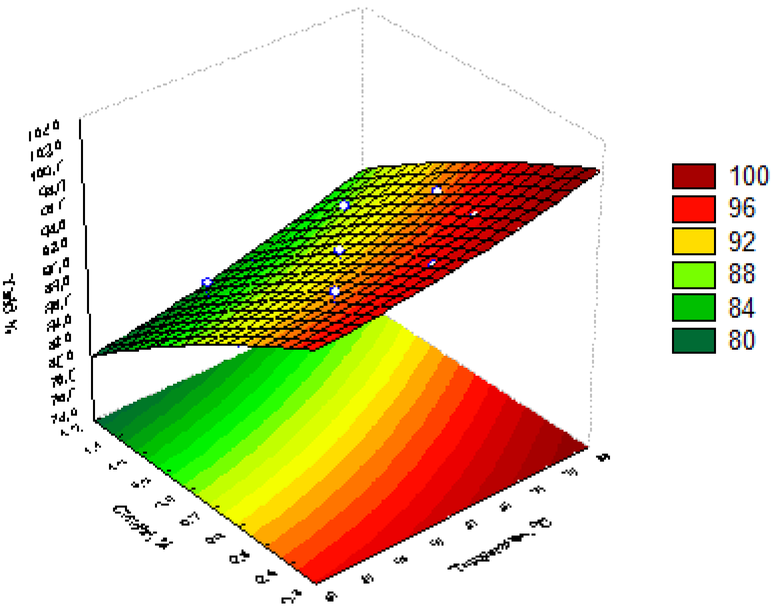

Fig. 3. Response surface profiles 3D of methyl ester yield

Figure 4 shows the optimum operating conditions for biodiesel synthesis, where the temperature and concentration of the catalyst are at the optimum conditions that produce yield maximum. In the figure, the optimum condition was obtained at a sodium hydroxide catalyst concentration of 0.396% and a reaction temperature of 74.142°C, yielding 98.736% with a reaction time of 20 minutes and the microwave method. Suwannapa and Tippayawon [13] that the optimum conditions for biodiesel production by microwave from beef tallow are methanol:oil ratio of 9:1, sodium hydroxide concentration of 0.62%, reaction time of 26 minutes, temperature of 60°C, the biodiesel yield is 99%. The transesterification reaction of vegetable oil using conventional methods, optimal conditions at molar ratio of methanol/oil of 6:1, Sodium hidroxyde catalyst concentration of 1%weight, reaction temperature of 65°C, mixing speed of 300 rpm and reaction time of 70 minutes obtained a yield of 97.5 % [20].

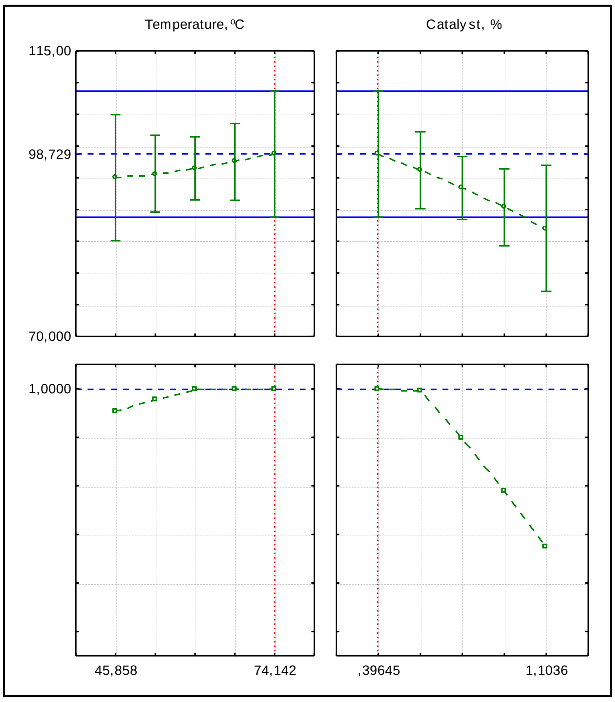

Fig. 4. Optimum operating conditions for biodiesel synthesis from beef tallow

4. Conclusions

The results of STATISTICA analysis showed that the catalyst concentration variable had a significant effect on the biodiesel synthesis process with p value = 0.00546. The results of optimization with the RSM method obtained the optimum yield with the amount of sodium hydroxide catalyst 0.396% and a temperature of 74.142ºC, with a biodiesel yield of 98.736%. Synthesis of biodiesel at mole ratio of methanol:oil (6:1), reaction time (20 minutes) using the microwave method. The form of the mathematical equation under optimum conditions is $y = 90.8107 - 8.7477 X_1$.

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