Application of Response Surface Methodology for Optimization of Biodiesel Production by Transesterification of Animal Fat with Methanol

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Abstract-In an effort to optimize the reaction conditions of biodiesel production from animal fat, response surface methodology was used. The combined effects of catalyst concentration, reaction time and methanol quantity of biodiesel yield were investigated and optimized using response surface methodology. A second order model was generated to predict biodiesel yield as a function of catalyst concentration, reaction time and methanol quantity by keeping the reaction temperature (55^{0}C to 60^{0}C) constant for all experiments A statistical model predicted the maximum animal fat methyl ester yield of 85.93% volume of oil at optimized parameters of methanol quantity (35% volume of oil), base catalyst concentration (0.46% weight of oil) and reaction time (90 minutes). Experimentally, maximum yield of 91% was obtained at the above parameters. A variation of 5.56% was observed between predicted maximum and experimental maximum yield.

Keywords- Transesterification, optimization, central composite design, response surface method.

1. Introduction

Currently, the cost of biodiesel is very high due to the use of crude and refined vegetable oil. Considerable research work is going on all over the world to find low cost sources in order to reduce the cost of biodiesel [1]. Reports indicate that source contributes 70% to 80% of the biodiesel cost. A few attempts have been made to use low cost sources such as animal fat, restaurant waste oil, etc. [1] [2] for the production of biodiesel. Animal fat is difficult to process because of its high Free Fatty Acid (FFA) content [3] [4]. Waste animal fats of tallow, lard and poultry fat can be collected from slaughter houses and meat processing companies. At the laboratory, they can be melted and filtered to obtain the fat and remove gum, protein residues, and suspended particles. Alkaline catalyzed transesterification process was used successfully to convert low FFA feed stock into biodiesel, However, this process cannot be used for high FFA feedstock because the yield decreases due to formation of soap [5] [6].

Biodiesel production from animal fat represent environmental friendly and lower price alternative. Currently, considerable study is going on to convert animal fat into biodiesel. Both esterification and transesterification reactions are being used to convert animal fat into respective esters. Huge amount of waste animal fat, tallow, lard, and poultry fat are available in slaughter houses and meat processing companies.

Some researcher used the MgO impregnated with a heterogeneous catalyst for esterification of tallow with methanol. In this process greater than 98% conversion of fat into biodiesel in 20 minutes is obtained. At 0.02% weight of moisture and free fatty acid 0.002% with methanol completely converted into biodiesel, but an additional 1 % weight of moisture resulted in soap formation [6] [7]. Considerable research work has done to use tallow, lard, and poultry fat for the production of biodiesel [6] [7] [8] [9]. In this work, an attempt has made to produce biodiesel from animal (goat) fat through the alkaline catalyzed transesterification process. Response surface methodology and central composite design methods are used to describe the effects of five level three factors and their reciprocal effects on biodiesel production.

2. Materials and Methods

The animal fat (Fig.1) has collected from meat processing companies and slaughter house within Mysore City (India). Analytical reagent (AR) methanol (99.3%) and sulphuric acid (99% pure) and NaOH in pellet form were purchased from the VASA Scientific Company, Bangalore. The fatty acid level animal fat was found at 3.5%. Table 1 shows the fatty acid composition of waste animal fat obtained from Gas Chromatography (GC-57) analysis, it has found that animal fat contains 49.36% saturated acid (Palmitic acid 21.23% and Stearic acid 28.13%), and 29.62% unsaturated acid (oleic acid27.33%, linoleic acid 2.29% and linolenic acid is zero). Saturated fatty acid chains are very important factors to find out the cold flow properties of waste animal fat oil as diesel oil.

Table 1 Fatty acid composition (%) of waste animal fat

Sl. NO	Components	Waste animal fat
1	Palmitic acid (16:0)	21.23%
2	Stearic acid (18:0)	28.13%
3	Oleic acid (18:1)	27.33%
4	Linoleic acid (18:1)	2.29%

The animal fat (waste) has high saturated free fatty acid ester and also high centane number and stability, more polyunsaturated reduce the cold point centane number and stability. The cold filter plugging point (CPFPP) of oil increases with an increase in content of saturated fatty acids.

2.1 Method used for production of biodiesel from waste animal fat

Single (alkaline base-catalyzed) stage transesterification process has used for the production of biodiesel as it free fatty acid level is less than (4%). Sodium hydroxide was used as a catalyst because it is cheaper and reacts faster than acid catalyst. Before the transesterification process of waste animal fat, it is heated at 110 to 120^0 C for one hour and then removes dust particle and impurities by filtering with cloth filter (Fig.2).

3. Experimental design

3.1 Parameter design methodology

In order to optimize the transesterification process parameter a five- level-three factors CCD has used. Three control parameters such as methanol (A) in ml, sodium hydroxide (B) in grams and reaction time (C) in minutes have selected for optimization of biodiesel yield. Each of the three control parameters has treated at five levels. Table 2 shows the code and un-coded levels of independent variables.

Fig.1 Waste animal fat

Fig.2 Boiling process

Table 2 Independent parameter and level used in CCD

Parameters	Units	Symbols	Levels				
			-1.68 (-α)	-1	0	1	+1.68 (+α)
CH_3OH (% v/ v of oil)	ml	A	26.6	30	35	40	43.4
NaOH(% w/ v of oil)	grams	B	0.464	0.6	0.8	1.0	1.136
Reaction Time	minute	C	39.6	60	90	120	140

3.2 Design of experiments

Table 3 shows the experimental matrix for CCD and biodiesel yield for the alkaline catalyzed transesterification process. Twenty experiments have conducted keeping the reaction temperature (55^0C to 60^0C) constant.

3.3 Experimental procedure

Initially the oil sample taken in the flat bottom flask with a 250ml capacity and temperature is maintained at 55^0C to 60^0C by a magnetic stirrer with hot plate. The sodium methoxide solution has prepared by dissolving required amount of sodium hydroxide in a required amount of methanol. Half of the prepared sodium methoxide solution has slowly added to the heated oil. Stirring and heating continued for 20 to 25 minutes.

Then remaining half of sodium methoxide solution has slowly added to the heated mixture. Stirring at low RPM (500 to 600) and heating up to 55^0C to 60^0C has continued up to reaction time. Similar procedure has used for all 20 experiments which have conducted according to the experimental matrix shown in Table 3. The mixture was allowed to settle overnight by gravity settling into clear, golden colour liquid biodiesel at the top layer and light brown glycerol at the bottom layers. Next day glycerol was drained off, leaving biodiesel and it was water washed two time. Pale yellow colour was stored in a bottle.

3.4 Statistical analysis

The experimental data were analyzed with XLSTAT for quadratic least square regression procedure using the equation. XLSTAT software has used in the regression analysis.

4. Results and discussion

Alkaline catalyzed transesterification process has conducted using the 20 samples with the free fatty acid level of 3.5%. In order to find the optimum conditions for maximum yield, experiments have conducted as per CCD experimental matrix shown in Table 3. Table 3 shows the observed yield of animal fat methyl ester. The multiple regression co-efficient have obtained by employing the least square method to predict a quadratic polynomial model for animal fat methyl ester yield. The regression model has found with the correlation coefficients of determination of R^2 having a value of 0.920 (Table 4). The predicted model for % of animal fat methyl ester yield in terms of code factors has given in equation 1

$$Y=24.689+13.041*A-13.172*B-8.603*C+9.354*A^2+13.699*B^2+7.884*C^2+12.686*A*B-0.531*A*C+3.37375*B*C \quad ....1$$

RSM was employed to understand the interaction of factors affecting animal fat methyl ester yield. Methanol quantity (β1) was the limiting factor and a small change in its value has greater effect on the yield. There was a significant interaction between methanol and catalyst quantity (β12) and catalyst quantity and reaction time (β23). There is a negative interaction between methanol and reaction time (β13).

A statistical model (equation no.1) has predicted that the maximum yield of waste animal fat methyl ester (85.93% volume of oil) has obtained at optimized parameters of methanol quantity (35% volume of oil), base catalyst concentration (0.46% weight of oil) and reaction time (90 minutes). Experimentally, maximum yield of 91% was obtained at above parameters. A variation of 5.56% was observed between predicted maximum and experimental maximum yield. This is there within the acceptable variation between theoretical and experimental value.

Fig.3 (a) and Fig.3 (b) show the 3 D response surface plot and 2D contour plots between quantity of methanol and catalyst concentration (NaOH) for different constant parameters. From Fig. 3(a) it can be seen that percentage of methyl ester yield has higher values for maximum methanol and minimum value of catalyst concentration at constant temperature and constant reaction time. Lower value of yield was obtained for a minimum value of methanol and maximum value of catalyst concentration at value of constant temperature and constant time.

Table 3 Experimental matrix for CCD and experimental result

Exp. No	A	B	C	CH_3OH (% v/v of oil)	NaOH (% w/v of oil)	Reaction Time(minute)	Yield (%)	
							Experimental	Predicted
1	-1	-1	-1	30	0.6	60	75	79.89
2	-1	-1	1	30	0.6	120	63	56.99
3	-1	1	-1	30	1.0	60	31	21.42
4	-1	1	1	30	1.0	120	22	12.02
5	1	-1	-1	40	0.6	60	79	81.66
6	1	-1	1	40	0.6	120	54	56.64
7	1	1	-1	40	1.0	60	75	73.20
8	1	1	1	40	1.0	120	75	62.42
9	0	0	-1.68	35	0.8	39.6	63	61.42
10	0	0	1.68	35	0.8	140	20	29.21
11	0	-1.68	0	35	0.46	90	91	85.93
12	0	1.68	0	35	1.13	90	25	41.28
13	-1.68	0	0	26.6	0.8	90	20	35.52
14	1.68	0	0	43.4	0.8	90	71	73.08
15	0	0	0	35	0.8	90	25	24.68
16	0	0	0	35	0.8	90	25	24.68
17	0	0	0	35	0.8	90	25	24.68
18	0	0	0	35	0.8	90	25	24.68
19	0	0	0	35	0.8	90	25	24.68
20	0	0	0	35	0.8	90	25	24.68

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Table 4 Regression coefficient of predicted quadratic polynomial model

Terms	Coefficients	Standard errors	Computed t-value	P-value
Linear				
β_0	24.689	4.04	6.12	0.000
β_1	13.042	2.68	4.87	0.001
β_2	-13.172	2.68	-4.92	0.001
β_3	-8.603	2.68	-3.21	0.009
Quadratic				
β_{11}	9.354	2.61	3.59	0.005
β_{22}	13.699	2.61	5.25	0.000
β_{33}	7.885	2.61	3.02	0.013
Interaction				
β_{12}	12.686	3.50	3.62	0.005
β_{13}	-0.531	3.50	-0.15	0.882
β_{23}	3.374	3.50	0.96	0.358
R^2	0.920			

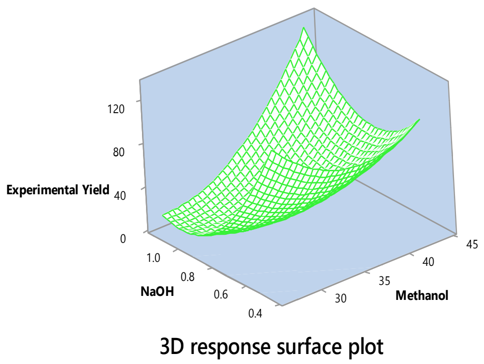

Fig. 3 (a) Response surface plot representing the Effect of catalyst concentration and quantity of methanol on experimental yield

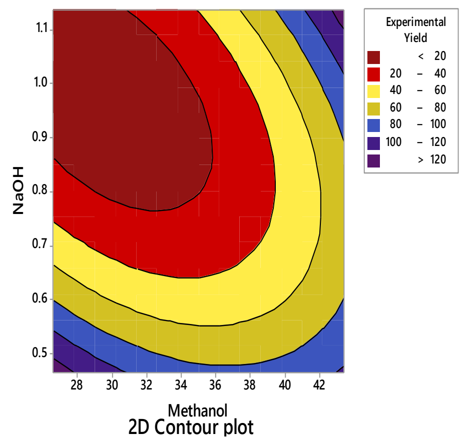

Fig. 3 (b) 2D Contour representing the Effect of catalyst concentration and quantity of methanol on experimental yield

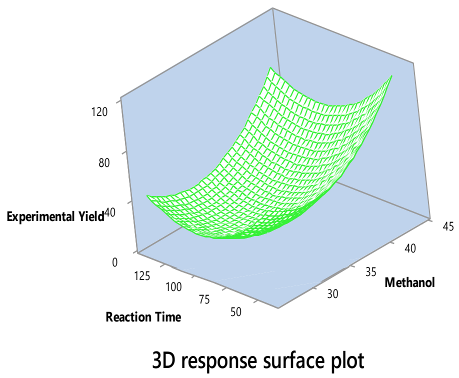

Fig. 4(a) Response surface plots represent the effect of reaction time and quantity of methanol on experimental yield

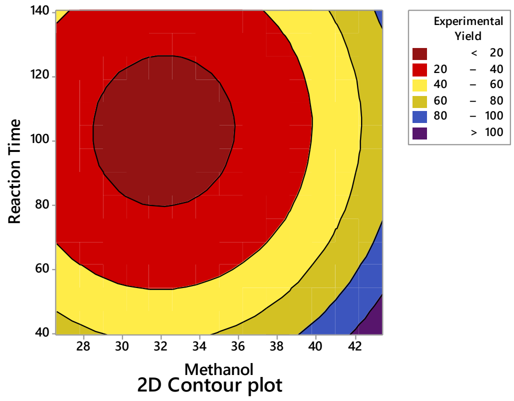

Fig. 4(b) 2D Contour plots represent the effect of reaction time and quantity of methanol on experimental yield

Fig. 4 (a) and Fig. 4 (b) show the 3 D response surface plot and 2D counter plots between quantity of methanol and reaction time for different constant parameters. From Fig. 4 (a) it can be seen that percentage of methyl ester has higher value of yield for higher values of methanol quantity and lower value of reaction time at constant temperature and constant catalyst concentration. Lower values of yield were obtained for lower values of methanol and higher value of reaction time at constant temperature and constant catalyst concentration.

Fig.5 (a) and Fig.5 (b) show the 3 D response surface plot and 2D contour plots between reaction time and catalyst concentration for different constant parameters. From Fig.5 (a) it can be seen that percentage of methyl ester has higher value of yield for maximum value of reaction time and a minimum amount of catalyst concentration at constant reaction temperature and constant methanol. Lower values of yield were obtained from a maximum value of catalyst concentration and minimum value of reaction time.

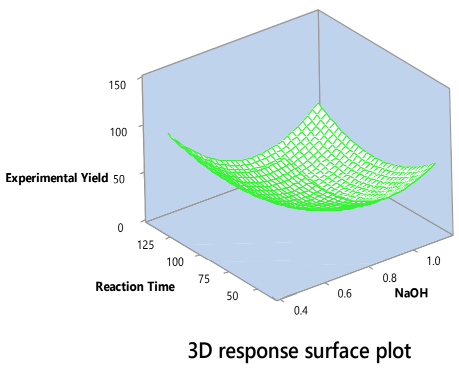

Fig. 5(a) Response surface plots representing the effect of reaction time and catalyst concentration on experimental yield

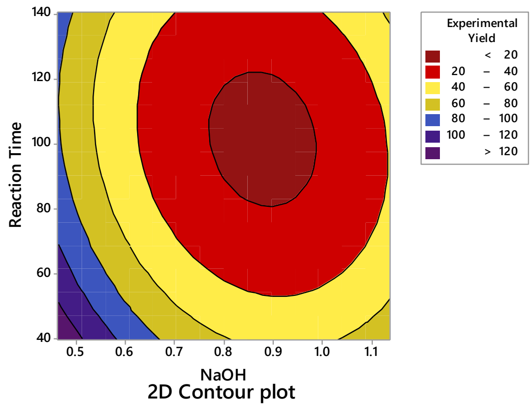

Fig. 5(b) 2D Contour plots representing the effect of reaction time and catalyst concentration on experimental yield

5. Conclusion

Based on the work carried out, it can be concluded that response surface methodology and central composite method can successfully be used to optimize the transesterification reaction parameters to maximize the biodiesel yield. Graphical 3D surface plots can be used to locate the optimum point. A full factorial central composite design was successfully used for experimental design and result analysis. It has predicted that the maximum yield of waste animal fat methyl ester (85.93% volume of oil) has obtained at optimized parameters of methanol quantity (35% volume of oil), base catalyst concentration (0.46% weight of oil) and reaction time (90 minutes). Experimentally, maximum yield of 91% was obtained at above parameters. A variation of 5.56% was observed between predicted maximum and experimental maximum yield. It can also be concluded that animal fat will become the potential source for the production of biodiesel.

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