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Central Composite Design and Desirability of RSM for Optimization of Biodiesel Production from Mixture of Animal Waste Fat Oil and used Cooking Oil

Swapna G. K., M. C. Math

***Abstract:** In this work, central composite design(CCD) and desirability approach of Response surface methodology (RSM) has been used for optimization of biodiesel yield produced from mixture of animal waste fat oil and used cooking oil (AWO) in the ratio of 1:1through alkaline transesterification process. In this work, methanol quantity, reaction time and sodium hydroxide concentration are selected as input parameters and yield selected as response. The combined effect of methanol quantity, reaction time and sodium hydroxide concentration were investigated and optimized by using RSM. The second order model is generated to predict yield as a function of methanol quantity, reaction time and sodium hydroxide concentration. A statistical model predicted the maximum yield of 96.9779% at 35ml methanol quantity (% v/v of oil), 75 min. reaction time and 0.6g (% wt./v of oil) of sodium hydroxide. Experimentally, the maximum yield of 97% was obtained at the above optimized input parameters. The variation of 0.02% was observed between experimental and predicted values. In this work, an attempt has also made to use desirability approach of RSM to optimize the input parameters to predict maximum yield. Desirability approach predicts maximum yield (97.075%) at CH_3OH (35.832% vol. /vol. of oil), NaOH (0.604 % wt./vol. of oil) and reaction time (79.054min.) was found for the AWO.*

***Keywords:** methanol, reaction time, sodium hydroxide, desirability, optimization, AWOME*

I. INTRODUCTION

All over the world search is going on to find better alternative to diesel oil because crude oil reserves are depleting at a faster rate[1][2][3]. In addition to this the price of crude oil is highly fluctuating in nature which affects adversely on the economy of oil importing countries like India [4][5][6]. The developing countries like India are heavily depending on import of crude oil to meet her energy requirements. The consumption of crude oil for energy requirements generates toxic gases which in turn degrades the breathing air quality.

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***** Correspondence Author
Swapna G. K.*, Research Scholar, Department of Thermal Engineering, VTU, RC, Mysuru, India.
M. C. Math, Associate Professor, Department of Thermal Power Engineering, Visvesvaraya Technological University, Center for Post Graduation Studies, Mysore, India.

India is the third largest importer of crude oil country in the world after China and the United States of America [7] during 2017-18. India has imported 213.932 million metric ton (MMT) and has invested Rs.4, 70,159 crores to import crude oil during 2016-17[7].

In India import of crude oil has increased 5.46% in quantity terms and 12.85% in value terms from 2015-16 to 2016-17. There is an

increase of 15.76% in import of crude oil from 2011-12 to 2016-17. To reduce the import of crude oil, oil importing countries are searching for a fuel which is similar to diesel oil.

Considering the problems associated with the use of diesel oil, efforts are going on to find an indigenous fuel Among many alternative fuels, biodiesel is considered as a best fuel for diesel engine because it can be produced from locally available renewable energy sources like edible, non edible vegetable oil, animal fat and used cooking oil[8][9][10][11].

Currently, cost of biodiesel is very high as compared to petro-diesel because it is produced from edible and non edible vegetable oils [12][13]. Search is going on to find suitable, low cost sources for the production of biodiesel [14]. In this direction animal waste fat oil and used cooking oil are considering as low cost sources because these sources are available at lowest price and moreover, these sources do not have commercial or resale value[15][16][17]. Currently, slaughter houses and big restaurants are facing lot of problems in disposing the animal waste fat and used cooking oil which are directly pumping into the drainages. This problem can be solved by using these waste sources for the production of biodiesel.

A team of researchers used animal waste fat oil for the production of biodiesel and results revealed that maximum yield(89%) was obtained at methanol (0.35 wt./wt.), reaction temperature ($62 \pm 1°C$), reaction time (2hr.) and by catalyzing with H_2SO_4(0.08 wt./wt.) and NaOH(0.01 wt./wt)[18].Waste tallow was used as a source for the production of biodiesel and results indicated that maximum yield of $99.01 \pm 0.71\%$ was obtained for chicken fat at reaction time (24hr.), temperature (50°C) and H_2SO_4(1.25g on fat wt. basis) and $93.21 \pm 5.07\%$ yield was obtained for mutton fat at reaction time (24hr.), temperature (60°C) and H_2SO_4(2.5g on fat wt. basis) respectively [19]. Two stage acid catalyzed processes were used to convert animal fat residues (AFR) from waste water into biodiesel.

In this work AFRs was treated with 5.4% (w/w) H_2SO_4 at a methanol/AFR ratio of 13:1 (50% w/w) and 60°C, resulting in 95% fatty acid methyl esters (FAMEs) with an acid value (AV) of 1.3 mg KOH/g biodiesel[20]. Evaluation of two purification methods of biodiesel production results in maximum yield of 76.8%,

90.8% and 91.5% (wt. /wt.) for chicken fat, beef tallow and pork lard respectively [21].

Biodiesel production from vegetable oil using heterogeneous acid and alkali catalyst obtained 96% yield at methanol-to-oil molar ratio 11.69:1, catalyst amount 2.52%, and reaction time 2.45hr.[22]. A study on production optimization and quality assessment of biodiesel from waste vegetable oil showed maximum yield of 96.15% using a methanol/oil molar ratio of 6:1, potassium hydroxide as catalyst (1%) and 65 °C temperature for one hour [23]. Co-solvent method is used to produce biodiesel form waste cooking oil with small pilot plant and results revealed the maximum yield of 98% at optimized process parameters: 1 wt. % potassium hydroxide catalyst, 20 wt. % acetone and 5:1 methanol to oil molar ratio, reaction temperature of 40°C and reaction time of 30 minutes [24]. Ultra sonication-assisted transesterification process is also used for production of biodiesel from waste cooking oil through heterogeneous ZnO Nano-catalyst and maximum yield of 96% was obtained at 1:6 volumetric oil-to-methanol molar ratio, 1.5 wt. % catalysts [25]. Response surface methodology was used for continuous biosynthesis of biodiesel from waste cooking palm oil in a packed bed reactor and maximum yield of 79% was obtained at the optimum conditions of 10.53 cm packed bed height and 0.57 ml/min substrate flow rate. The optimum predicted fatty acid methyl ester (FAME) yield was 80.3% [26]. Efforts are made to use high FFA feedstock such as waste cooking oil with an acid value of 68.81 mg KOH/g for the production of biodiesel through the use of two stage transesterification process. In the first stage, the source is treated with 20% methanol to oil ratio (by volume), 0.4 vol. % H_2SO_4 at 50 °C. The acid value reached 3.76 mg KOH/g after 5hr. of reaction time. In the next step, acid value was brought down to 0.86 mg KOH/g from 3.76 mg KOH/g after treating the product of first step with NaOH concentration 0.5 N, excess alkali 15% at 60 °C temperature and 40 min. reaction time[27]. Use of response surface methodology for process optimization of biodiesel production from mahua oil results in maximum yield of 98% at 0.7% KOH and 0.25 v/v CH_3OH[28]. Efforts are also made to use microwave assisted transesterification process to convert rice bran oil into methyl ester, resulting in maximum yield of 93.8% at power of 160W [29]. Application of response surface methodology for optimization of biodiesel production by transesterification of soybean oil with ethanol, results in maximum yield of 95% at optimum values of molar ratio(9:1), catalysts(1.3wt .%), time(80min.) and temperature (40°C)[30]. A statistical analysis has been carried out which predicts the maximum yield (73.58%) at optimized values of methanol quantity (40% volume of oil), sodium hydroxide concentration (1.0% wt. of oil) and reaction time (75min.). The experimental yield is 82.1% is obtained at the above optimized values by using garcinia gummi gutta oil [31].

Response surface methodology is a good method for estimating the number of experiment required for the production of biodiesel [32]. It also helps to identify the maximum operating condition. Desirability approach is also another method of obtaining maximum yield at maximum input parameters [33].

Novelty of this work is the use of mixture of animal waste fat and used cooking oil (AWO) in the ratio of 1:1 as a source for biodiesel production. In addition to this along with central composite design, desirability approach of RSM has been used to optimize transesterification process parameters to maximize the yield.

II. MATERIALS AND METHODOLOGY

The animal waste fat has collected from meat processing companies and slaughter house .The used cooking oil(UCO) is collected from hotel, catering, restaurant and potato chip maker within the Mysuru city(India).

Table 1 shows the fatty acid distribution of used cooking oil (UCO), animal waste fat oil (AFO) and mixture of animal waste fat oil and used cooking oil (AWO) in the ratio of 1:1 obtained from Gas Chromatography (AOAC -996.06) analysis. It has found that AFO, UCO and AWO contain 58.02%, 13.95% and 49.96% saturated acid (Palmitic acid and Stearic acid) respectively. It also found that AFO, UCO and AWO contain 28.76%, 81.41% and 27.5% unsaturated acid (Oleic acid, Linoleic acid and Linolenic acid) respectively. It also found that AFO, UCO and AWO have fatty acid level of 1.41 %, 1.09 % and 0.62 % respectively as shown in Table 1.

Table1: Fatty acid composition of AFO, UCO and AWO

Sl. No.	Components	AFO	UCO	AWO
1	Palmitic acid(16:0)	29.89%	9.6%	20.87%
2	Stearic acid (18:0)	28.13%	4.35%	29.09%
3	Oleic acid (18:1)	27.44%	27.91%	27.5%
4	Linoleic acid (18:1)	0.21%	53.5%	< 0.1
5	Linolenic acid (18:1)	1.11%	0	< 0.1
6	FFA	1.41%	1.09%	0.62%

A. Experimental Procedure

Fig.1 shows the transesterification method used for production of methyl ester (biodiesel) from AWO. Table 2 shows five levels three factors code and un-coded levels of independent variables used in CCD. Experimental matrix to optimize the yield of AWOME.

Table 2: Independent parameter and levels used in CCD (RSM)

Name	Units	Symbols	Levels				
			-1.68 (-α)	-1	0	1	+1.68 (+α)
CH_3OH (% v/v of oil)	ml	A	26.59	30	35	40	43.41
NaOH (% w/v of oil)	gm.	B	0.2636	0.4	0.6	0.8	0.9364
Reaction Time	min.	C	49.77	60	75	90	100.23

B. Design of Experiments

Table 3 shows experimental matrix used for central composite design which is generated using design expert software D11 version. The twenty experiments were carried out as per experimental matrix(Table 3) and experiments were conducted as per the transesterification process flow diagram shown in Fig.1by keeping reaction temperature constant (55 – 60°C).

Plate 1 show the yield obtained during 20 experiments.

Table 3: Experimental matrix to optimize the yield of AWOME

Sl no.	CH_3OH (ml)	NaOH (gram)	Reaction Time (min)	AWOME	
				Expt. yield(%)	Predicted yield(%)
1	30	0.4	60	32	32.35162
2	40	0.4	60	64	64.43565
3	30	0.8	60	63	63.01578
4	40	0.8	60	59	59.09981
5	30	0.4	90	49	48.80867
6	40	0.4	90	74	73.8927
7	30	0.8	90	58	57.47283
8	40	0.8	90	47	46.55686
9	26.59104	0.6	75	42	42.16464
10	43.40896	0.6	75	60	59.96479
11	35	0.263641	75	43	42.66533
12	35	0.936359	75	45	45.4641
13	35	0.6	49.7731 1	78	77.41904
14	35	0.6	100.226 9	80	80.71039
15	35	0.6	75	97	96.97779
16	35	0.6	75	97	96.97779
17	35	0.6	75	97	96.97779
18	35	0.6	75	97	96.97779
19	35	0.6	75	97	96.97779
20	35	0.6	75	97	96.97779

Plate 1: Twenty samples of AWOME

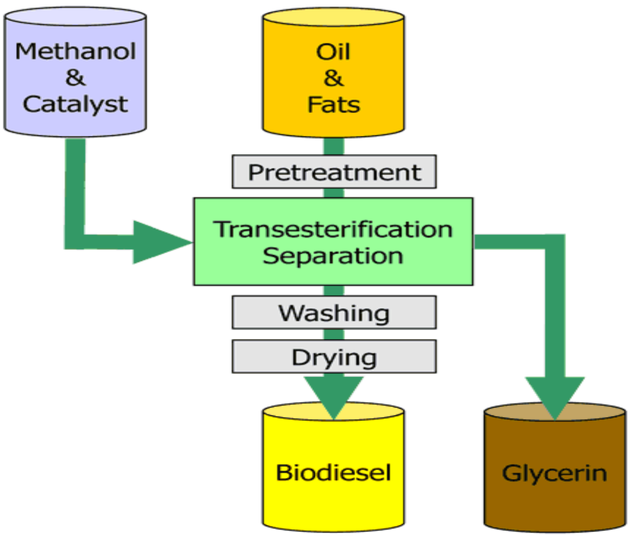


Fig. 1: Process Flow Diagram of Biodiesel Production

C. Statistical analyses

Design expert D11 software is used for analyzing the experimental data for quadratic least square regression procedure using the equations (Eq.1). In this work coefficients have been obtained by using multi regression analysis. The correlations have been developed in order to predict the output (yield). By using statistically significant model, the correlation between transesterification process and yield has been obtained. Finally desirability approach of RSM is used to get the optimum values of CH_3OH, NaOH and reaction time.

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

Where, Y is the response (dependent variables), β_0 the constant coefficients, β_{ii} and β_{ij} the coefficients for linear, quadratic and interaction effect, x_i and x_j the factors (independent variables) and ε is the error.

III. RESULTS AND DISCUSSION

A. RSM model analysis

The experimental results have been fitted to second order polynomial equation using design expert software D11 version. The quadratic model for the output has been developed in terms of actual parameters and are shown in Eq. (2)

$$\text{Yield} = (96.98 + 5.29A + 0.8321B + 0.9785C - 9.00AB - 1.75AC - 5.50BC - 16.23\,A^2 - 18.71B^2 - 6.33C^2) \qquad (2)$$

Where A is the CH_3OH in ml, B is NaOH in gram and C is the Reaction time in min.

The equation in terms of coded factors can be used to make predictions about the yield for a given levels of each factor. By default, the high levels of the factors are coded as +1 and the low levels are coded as -1. The coded equation is useful for identifying the relative impact of the factors by comparing the factor coefficients. Positive sign (A, B and C) indicates synergistic effect and negative sign (AB, AC, BC, A^2, B^2, C^2) indicates antagonistic effect on Biodiesel yield.

ANOVA was used for the principal model analysis that gives information for the P value. Table 4 shows ANOVA for yield. The model is significant because the value of P is found less than 0.05. From Table 4, it is clear that CH_3OH (ml), NaOH (gram) and Reaction time (min.) are the most influencing input parameters and play an important role on yield.

Table 4: ANOVA for yield indicating value of P

Source	Yield
Model	< 0.0001
A	< 0.0001
B	0.0048
C	0.0024

Table 5 shows precision index values for yield. From Table 5, it is clear that difference between the values of Predicted $R^2$and Adjusted R^2 is less than 0.2 for yield, which indicates that Predicted $R^2$is in reasonable agreement with the Adjusted R^2.Adeq.precisionmeasures the signal to noise ratio.

A ratio greater than 4 is desirable. It is clear from Table 5, that Adeq. precision is greater than 4 for yield.

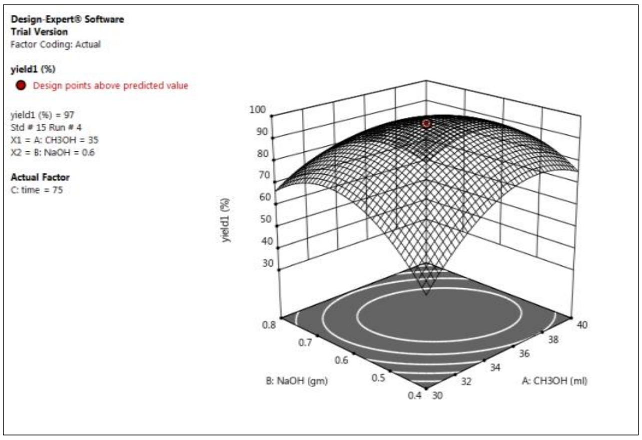
This means model can be used to navigate the design space. From the values of Press presented in Table 5 shows that model prediction is very close to experimental value.

Table 5: Precision index values

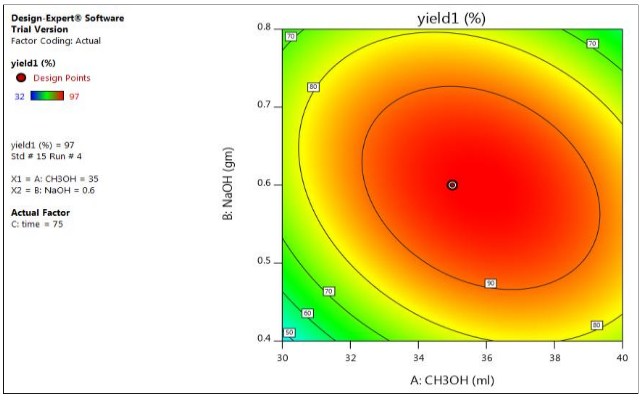
Source	Yield
Standard Deviation	0.6394
Mean	59.40
C.V. %	1.08
Model Degree	Quadratic
R^2	0.9995
Adjusted R^2	0.9986
Predicted R^2	0.9954
Adeq Precision	123.7802
Press	19.23

B. Interactive effect of CH3OH, NaOH and Reaction Time on Yield

Fig. 2 (a) and (b) show the interactive effect of CH_3OH and NaOH on yield on surface plot and counter plot respectively. Optimum yield for AWOME (97%) was obtained at 35ml CH_3OH and 0.6g NaOH and 75min. reaction time. From Fig. 2(a) and (b) , it is clear that yield increases, reaching maximum value and then decreases with the increase in CH_3OH for a given NaOH. This may be due to the fact that higher quantity of methanol will have negative effect on yield for a given catalyst concentration. From Fig.2 (a) and (b), it is also clear that yield increases, reaching maximum value and decreases with the increase in NaOH concentration for a given CH_3OH. This may be due to the formation of soap at higher concentration of catalyst for a given methanol quantity.



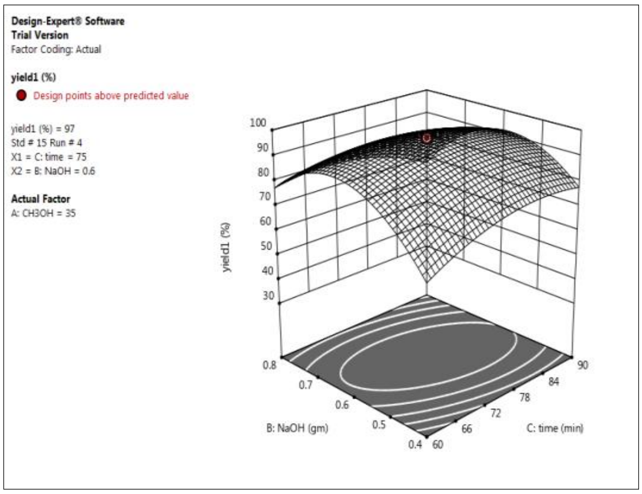
(a)



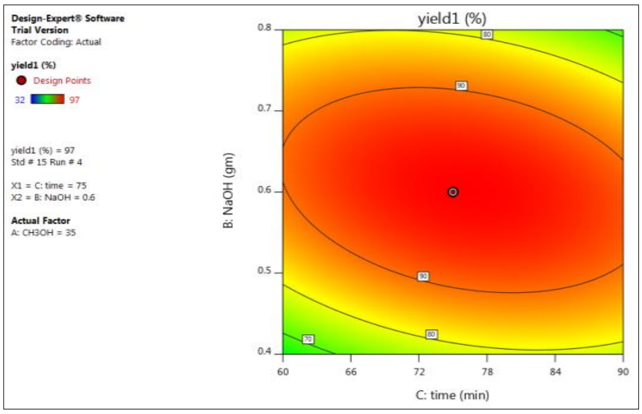
(b)

Fig. 2: (a) 3D Response surface plot and (b) 2D Counter plot of effect of CH_3OH and NaOH on yield for AWOME respectively

Fig. 2 (c) and (d) show the interactive effect of reaction time and NaOH on yield on surface plot and counter plot respectively. Optimum yield for AWOME (97%) was obtained at 35ml CH_3OH and 0.6g NaOH and 75min. reaction time. From Fig. 2(c) and (d), it is clear that yield increases, reaching maximum value and then decreases with the increase in reaction time for a given NaOH concentration. This may be due to the fact that the reaction may be reached equilibrium hence there will be a reverse reaction to occur. In addition to this, there is an increase in the probability of biodiesel hydrolysis under alkaline condition. From Fig.2 (c) and (d), it also clear that yield increases, reaching maximum value and decreases with the increase in NaOH concentration for a given reaction time. This may be due to the formation of soap at higher concentration of catalyst for a given reaction time.



(c)

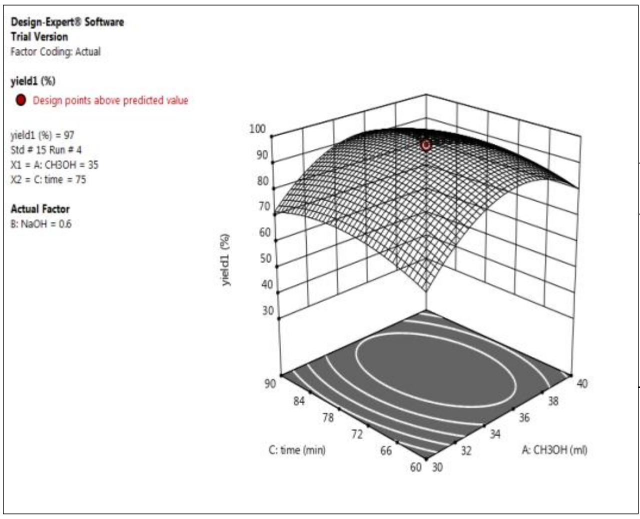


(d)

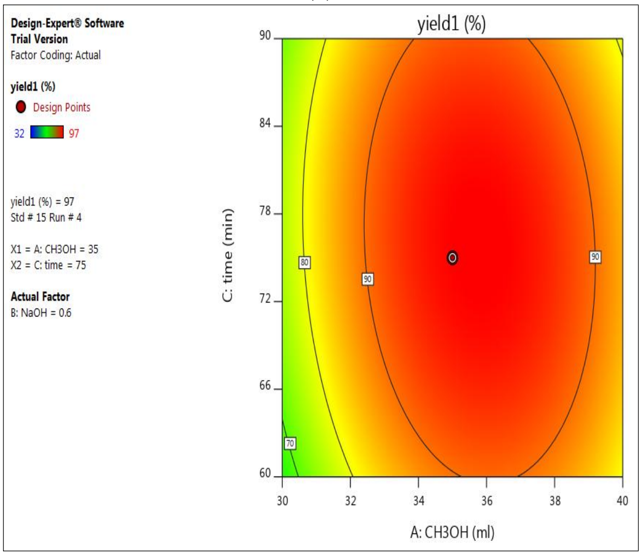
Fig. 2:(c) 3D Response surface plot and (d) 2D Counter plot of effect of NaOH and reaction time on yield for AWOME respectively

Fig. 2 (e) and (f) show the interactive effect of CH_3OH and reaction time on yield on surface plot and counter plot respectively. Optimum yield for AWOME (97%) was obtained at 35ml CH_3OH and 0.6g NaOH and 75min. reaction time. From Fig. 2(e) and (f), it is clear that yield increases, reaching maximum value and then decreases with the increase inCH_3OH for a given reaction time.

This may be due to the fact that higher concentration of methanol will create problem in separating ester from glycerol for a given reaction time. With the increase in reaction time for a given CH_3OH, the variation of yield found to be followed the above trend.



(e)



(f)

Fig.2 (e) and (f) 3D Response surface plot and 2D Counter plot of Effect of CH_3OH and reaction time on yield for AWOME respectively.

C. Optimization using desirability of RSM

In this work along with CCD, desirability approach of RSM has been used to find optimal transesterification process parameters for optimization of yield. Table 6 shows range of input parameters and AWOME yield. Yield has lower limit and upper limit and goal as shown in Table 6. Every input parameter also has lower limit, upper limit and goal. Table 7 shows optimization result of desirability approach of RSM. From Table 7, it is observed that maximum desirability of 1.000 was obtained at the input parameters like 35.832CH_3OH (% vol. /vol. of oil), 79.054min. reaction time and 0.604 catalyst concentration (gram/ % vol. of oil) and these could be considered as the optimum parameters for maximum yield (97.075%). Fig. 3 shows the contour plot for desirability for the predicted model which revealed that the overall desirability value is higher in region where there is optimum input values.

D. Validations of Test Results

In order to validate the optimized reults, the experiments were conducted thrice at the input parameters of 35.832 ml CH_3OH, 0.604g NaOH and 79.054min reaction time for 100ml of AWO. For the actual yield, the average of all the three test reults were calculated. Table 8 shows the validation of test results. From Table 8, it is clear that percentage of error was very small, which indicate that predicted values is in good agreement with experimental values.

Table 6: Range of AWOME input parameters and yield

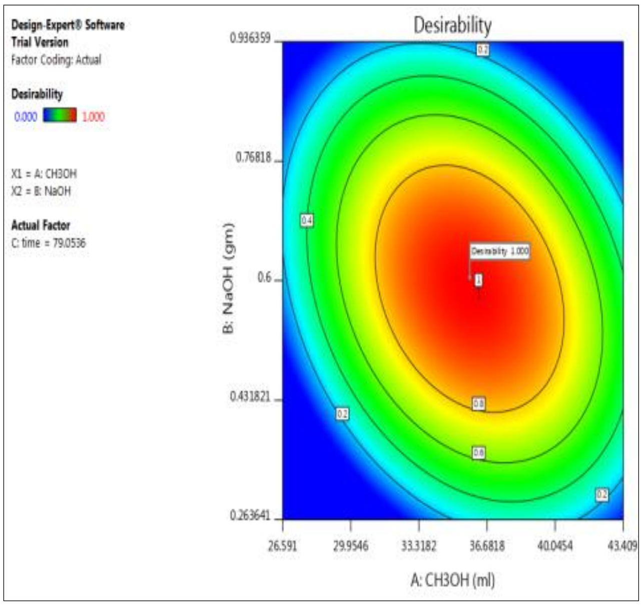
Name	Goal	Lower Limit	Upper Limit	Lower wt.	Upper wt.	Importance.
CH_3OH	is in range	26.591	43.409	1	1	3
NaOH	is in range	0.263641	0.936359	1	1	3
Time	is in range	49.7731	100.227	1	1	3
Yield	maximize	32	97	1	1	3

Table 7: Optimization result of desirability of RSM

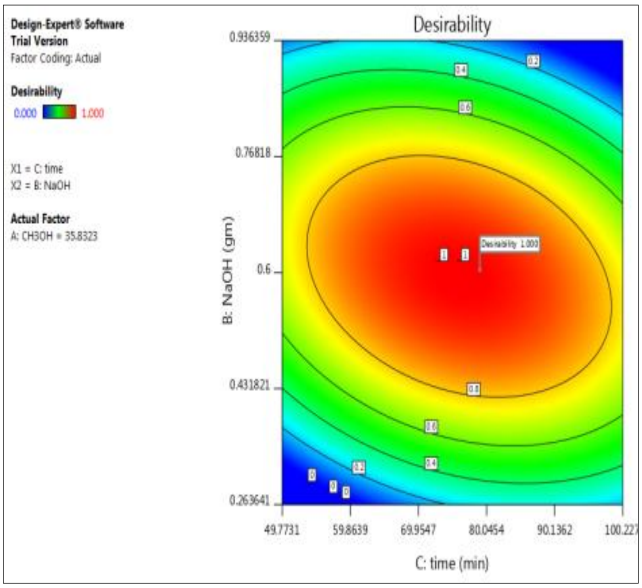
CH_3OH (ml)	NaOH (gram)	Reaction Time(min.)	Yield (%)	Desirability	
35.832	0.604	79.054	97.075	**1.000**	Selected

Table 8: Validation of test results

CH_3OH (35.832 ml), NaOH(0.604g) and reaction time(79.054min.)			
	Predi.	Expt.	Error (%)
Yield (%)	97.075	97.091	0.016



(a)



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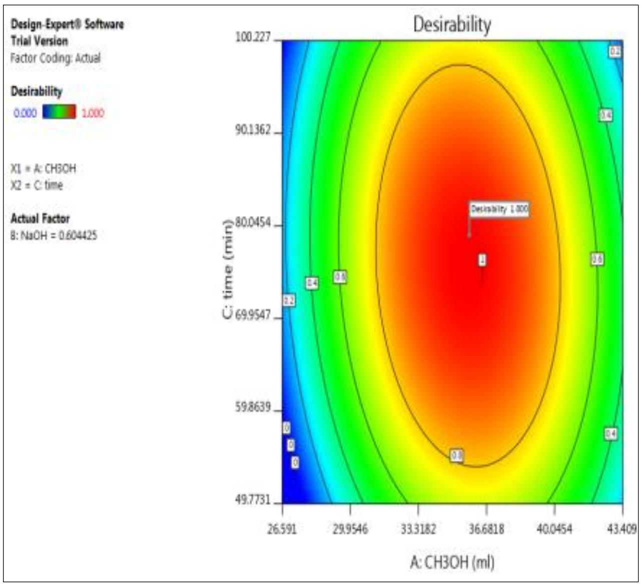

(c)

Fig.3: (a), (b) and (c) 2D Contour plot of desirability on Yield for AWOME

E. Fuel Properties of mixture of animal waste fat oil and used cooking oil

Table 9 shows physical properties of AWO which have been found out by using IS standard methods.

Table 9: Physical properties of AWO, AWOME and Diesel oil.

Fuel properties	unit	diesel	AWO	B100(AWOME)
Density	kg/m^3	810	900	836.7
Kinematic Viscosity at 40 °C	cSt	4.13	33.74	4.93
Flash Point	°C	52	263	106
Fire Point	°C	58	278	114
Cloud Point	°C	-3	19	9
Pour Point	°C	-14	13	2
Calorific Value	MJ/kg	42.7	28.4	34.2

From Table 9, it is clear that AWO and AWOME are denser than diesel oil. AWO (33.74cSt) is more viscous than AWOME (4.936cSt) and diesel oil (4.13cSt). AWO and AWOME have 33.4895% and 19.9063% respectively lower calorific value than diesel oil. The AWO and AWOME have higher flash and fire point than diesel oil. However, both AWO and AWOME have poor cloud and pour point than diesel oil. Analyzing these properties of AWO indicates that direct use of this source will hamper the hardware of diesel engine and may cause poor performance.

F. Uncertainity analysis of measuring instruments

Table 10 shows uncertainity analysis carried out for fuel properties measuring instruments in order to justify the experimental values of fuel properties.

Table 10: Uncertainty analysis

Instrument	Measured Parameter	Uncertainty (%)
Density meter	Density at 40°C	±0.21
Redwood viscometer	Dynamic viscosity at 40°C	±0.03
	Kinematic viscosity at 40°C	±0.34
Automatic cloud point system	Cloud point	±0.2
Pensky Martins closed cup apparatus	Flash point	±0.2
Automatic cloud point system	Pour point	±0.2
Bomb calorimeter	Calorific value	±1.3

IV. CONCLUSIONS

The following important conclusions have been drawn from the above study:

- Central composite design and desirability approach of RSM are found powerful tool to optimize the transesterification process parameters to find maximum biodiesel yield.
- It is found that methanol quantity, catalyst concentration and reaction time have greater influence on biodiesel yield.
- Higher concentration of methanol, catalyst concentration and longer reaction time beyond the optimal values have negative effect on biodiesel yield.
- The variation of 0.02% was observed between experimental and predicted yield.
- Desirability approach used for optimization of transesterification process parameters is found very simple and efficient tool.
- A high desirability of 1.000 was obtained at the optimum input parameters of CH_3OH(35.832ml), NaOH (0.604g) and reaction time (79.054min.). The maximum predicted yield is 97.075%.

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AUTHORS PROFILE

She is Research Scholar perusing her Ph.D in the Thermal Engineering at VTU,RC, Mysuru (India). She obtained her M.Tech in the Department of Thermal Power Engineering at VTU,PG Center, Mysore (India). She obtained her B.E. from Sri Jaya Chamarajendra Engineering College (SJCE), Mysore (India). She published four papers in international journals and national conferences.

He is working as Associate Professor in the Department of Thermal Power Engineering at Visvesvaraya Technological University, center for post graduation studies, Mysore. He obtained his Ph. D in IC engines and biodiesel from Jawaharlal Nehru Technological University, Hyderabad (India) in 2013. He did his M.Tech in Thermal Power Engineering from Visvesvaraya Technological University, Belgaum (India) in 2006 and B.E in Mechanical Engineering from Karnataka University, Dharawar (India) in 1996. He published 46 technical papers in International / national journal and conferences. He authored 8 textbooks on thermal engineering. He guided three international projects on super mileage vehicles which were participated in Shell Eco-Marathon competition held at Malaysia and Manila during 2011, 12 and 13. He is a life member of ISTE. He is guiding eight research scholars in the area of dual fuel and flexi fuel vehicle and alternative fuels.