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Optimization of biodiesel production from waste frying palm oil using definitive screening design

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Abstract

Research on alternative fuels is increasing these days due to the fact that these fuels can be afforded without any climate-change aggravation. Biodiesel from different feed stocks has a potential to replace the existing diesel fuel, but the cost involved in the production of biodiesel is more. To mitigate the high production cost, low cost feed stocks of used cooking oils (UCO) can be used. As per the latest statistics approximately 198 MMT/yr of edible oil is used around the world. These UCO are dumped after usage which causes environmental imbalance. In this research an attempt was made to utilize the waste frying palm oil (WFPO) which was available in the college for biodiesel production. To maximize the biodiesel yield latest optimization technique of definitive screening design (DSD) was implemented. It was concluded that maximum yield of 96.23% was achieved at molar ratio of 6:1, reaction temperature of 55^0C and the catalytic concentration of 1wt%. The fuel samples were tested for different physiochemical properties and fatty acid compositions. It was observed that the properties were within the limits of international standards. Hence, by using the WFPO the overall production cost was reduced and optimization by DSD enhanced the biodiesel yield.

Keywords: Waste fried palm biodiesel, optimization, definitive screening design, fatty acid composition, soap

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1. Introduction

To improve the economy of any country energy plays a key role. Petroleum products are the major commercial source of energy across the world. In India the demand for the diesel is five times higher than petrol (Dhar et al., 2012; Kolakoti and Rao, 2015). As per the latest population reports, India needs to generate 4 to 5-fold more energy than the present. In order to meet the demand, one must rely on the commercial sources which are finite and they are in verge of depletion (Agarwal and Rajamanoharan, 2007). Apart from the availability of the fossil fuel, their applications in internal combustion engines also increase the air pollution levels by emitting harmful exhaust tail pipe emissions like NOx, CO, UHC, CO_2 and smoke etc. (Modi et al., 2007; Talamala et al., 2016; Rao and Rao, 2017; Kolakoti and Rao, 2017c). To mitigate the challenges of exhaust emissions, biodiesel from edible and nonedible oils is best the possible solution. Researches and demonstrated project reveal that the alternative fuels from edible and non-edible oils can reduce the harmful exhaust emissions to a greater extent (Kalam et al., 2011). The unique properties in biodiesel can improve the combustion propensity (Demirbas, 2005), biodiesel acts as a best lubricant and the molecular oxygen presence helps in improved combustion with limited emissions (Kolakoti and Rao, 2017a). By implementing biodiesel in diesel engines will reduce the reliance on foreign oil products and increase the economy of a country like India. The raw oils which are used for the production of biodiesel possess high viscosity. This high viscosity can be reduced by following pyrolysis, thermal cracking, preheating and transesterification. Among the mentioned processes transesterification is a widely used method (Kolakoti and Rao, 2017b). During transesterification process several process parameters will influence the biodiesel yield some of them were catalytic concentration, methanol to oil ratio, reaction time and reaction temperature. Producing biodiesel from plant oils

including the cost of catalysts, alcohol and processes will turn out to be more expensive than diesel, to overcome this problem cooking oils which are dumped after several uses, which are of no further use can be converted into biodiesel.

In order to achieve the maximum biodiesel yield with limited raw material cost and time, optimization techniques are most suitable (Wu et al., 1999; Vicente et al., 2007; Cavalcante et al. 2010). The advanced optimization technique of definitive screening design was implemented (DSD). The DSD is a new optimization technique in Minitab-18 it is similar to factorial design and it is more time efficient and it can determine the main effects and contributions of the factors early in the analysis. By implementing this optimization technique, the biodiesel yield can be improved, on the other hand the important contributing factors for biodiesel yield were also be analyzed.

2. Materials and Methods

2.1 Materials

Fried palm oil of 10 liters was collected from the different food courts, Methanol, Sodium hydroxide, Sulphur acid was purchased from the Sigma-Aldrich chemical company. Different sizes of glass beakers, RPM controlled electro-mechanical stirrer, Distilled water were used for the biodiesel production.

2.2 Experimental procedure

Waste frying palm oil (WFPO) which was collected from our college was first preheated and then filtered with surgical cotton to remove the dust and unwanted suspended particles. To reduce the high viscosity of the palm oil a widely used transesterification process was followed. In this transesterification process the exchange of organic group of esters with organic group of alcohol (CH_3OH), which are catalyzed by addition of base catalyst (NaOH) to form as methyl esters. Sodium hydroxide pellets of 4 grams were mixed with 90ml of methanol to form a methoxide solution and it was added in 500ml of esterified WFPO and the solution was maintained at desired reaction temperatures (27^0C to 55^0C). After a period of time (60 to 180min) there will be a clear separation of glycerin and methyl ester, the heavy density glycerin gets settled in the bottom as shown in the Figure 1 was removed and the methyl esters was washed continuously with distilled water until a clear separation of oil and water appears as shown in the fig.2. The obtained fuel samples were tested for different physicochemical properties by following the international standards as shown in the Table 1. Biodiesel yield obtained from transesterification process depends on factors like reaction temperature, reaction time, molar ratio and catalytic concentration by variation in the amounts of these factors' biodiesel yield can be modified. In order to achieve the maximum yield, in our investigation definitive screening design in design of experiments was used for optimization of biodiesel for producing highest possible yield.

Figure 1. Glycerin and Methyl ester separation

Figure 2. Methyl ester and water separation

Table 1. Characterization of WFPO methyl ester

S.No	Properties	Diesel	WFPO	Standards
1	Viscosity (cSt)	2.8	4.2	ASTM-D445
2	Density (kg/m^3)	830	870	ASTM-D1298
3	Flash point (^{o}C)	62	145	ASTM-D975
4	Cloud point (^{o}C)	-12	-3 to 10	ASTM-D975
5	Pour point (^{o}C)	-16	-5 to 7	ASTM-D975
6	Cetane number	47	53	ASTM-D4737
7	Calorific value (MJ/kg)	44	38	ASTM-D2382

2.3 Definitive Screening optimization

Definitive screening design (DSD) is a new optimization technique in the Design of Experiment (DOE) MinTab-18. It identifies the most important factors early in the experimentation process. Definitive screening designs can fit 2–48 factors. This method identifies the important key parameters which influence the output for a given input. It also reduces the number of input parameters compared to other standard techniques like full factorial, response surface and mixture design etc. This technique is most suitable for minimum variable parameters; it also allows the user to restrict the input variable process parameter to a particular limit. Definitive screening is useful when the user wants to consider both linear and quadratic terms and to identify the

most critical factors Due to the wide range of advantages in definitive screening technique in this research, we adopted a two-level fractional factorial design with three factors. The three contributing factors were molar ratio (A), catalytic concentration (B) and reaction temperature (C) and their ranges are shown in the Table 2. The catalytic concentration (NaOH) was varied from 3.50 to 5.0, Molar ration (Methanol to oil ratio) 4:1 to 6:1 and the reaction temperature was varied from room temperature (27^0C) to methanol evaporation temperature (55^0C) and the catalytic concentration of 3 to 5 grams. (Catalytic concentration is the weight percent of catalyst taken to the amount of waste fried palm oil used i.e. 3.5 grams of NaOH in 500 ml of oil is equal (3.5/500)*100 =0.7wt%)

Table 2. Process parameters and their ranges

S.No	Variable	Range Minimum	Range Maximum
1	Reaction Temperature ($^{\circ}$C)	27	55
2	Catalytic Concentration (grams)	3.5	5
3	Molar Ratio	4:1	6:1

Table 3 represents the matrix for the three contributing factors generated by DSD and yield was chosen as a response. Based on the system generated matrix, 13 experiments were conducted and the response yield was obtained. The yield of the biodiesel is calculated from the Eq.1.

$$\text{Yield (\%)} = \text{Weight of biodiesel/Weight of raw oil} \quad (1)$$

Table 3 Matrix of process parameters and their response yield

Run No	Reaction Temperature	Catalytic Concentration	Molar Ratio	Yield (%)
1	55	5	5:1	93.24
2	27	4.25	4:1	84.97
3	41	3.5	4:1	84.85
4	41	4.25	5:1	88.32
5	55	3.5	4:1	86.55
6	27	3.5	5:1	86.72
7	41	5	6:1	93.95
8	27	5	6:1	93.15
9	27	3.5	6:1	90.10
10	27	5	4:1	86.79
11	55	5	4:1	90.61
12	55	3.5	6:1	93.82
13	55	4.25	6:1	94.40

2.4 Analysis of Variance

Analysis of variance (ANOVA) is based on an approach, in which the procedure uses a variance to predict whether the means are differentiable. It also measures the importance of contributing factors by comparing the response (Yield) variable at different levels. Based on the experimental yield matrix the response yield was analyzed and the ANOVA Table 4 was generated for the contributing factors.

Table 4. Analysis of Variance

S.No	Source	DF	Adj SS	Adj MS	F- Value	P-Value
1	Model	3	153.333	51.111	111.41	0.000
2	Linear	3	153.333	51.111	111.41	0.000
3	Reaction Temperature	1	28.527	28.527	62.18	0.000
4	Catalytic Concentration	1	24.665	24.665	53.76	0.000
5	Molar Ratio	1	100.141	100.141	218.27	0.000
6	Error	9	4.129	0.459		
7	Total	12	157.462			

The contribution of each factor can be found out by using p and t values, the lower the p value better the significance of the contributing factor, from the Table 5, the value of p can be found 0.000 in all the cases which indicates the design was very suitable, t value indicates the individual contribution of each factor to the response. From the Table 5, it is clear that molar ratio has the highest contribution in the yield, the t value of molar ratio is (14.77).

Table 5. Definitive screening analysis

S.No	Term	Coef	SE Coef	T-Value	P-Value	VIF
1	Constant	89.806	0.188	478.05	0.000	
2	Reaction Temperature	1.689	0.214	7.89	0.000	1.00
3	Catalytic Concentration	1.571	0.214	7.33	0.000	1.00
4	Molar Ratio	3.165	0.214	14.77	0.000	1.00

In addition, Table 6 represents the Standard deviation (S) Coefficient of determination(R-sq) and adjust coefficient of determination(R-sq(adj)). S represents the standard deviation between data values and fitted values. Lower the S values better the model describes the response. The R-sq represents the percentage of variation in the responses and it also determines how well the model fits the experimental data. Higher R-sq indicates that *most of the variations* in response can be explained, here R-sq (97.38%) indicates that the model fits the data.

Table 6. R-sq values for the optimized model

S.No	S	R-sq	R-sq (adj)	R-sq(pred)
1	0.677335	97.38%	96.50%	95.43%

2.5 Pareto chart of standardized effects

The Pareto chart gives the information about the absolute values of the standardized effects from maximum to minimum effect. The standardized effects test the null hypothesis. The chart also plots a reference line which indicates the effects that are statistically significant and it is denoted by alpha. From the fig.3 all the three contributing factors cross the reference line which indicates that the all the three factors statically significant with 95% confidence and the null hypothesis can be rejected.

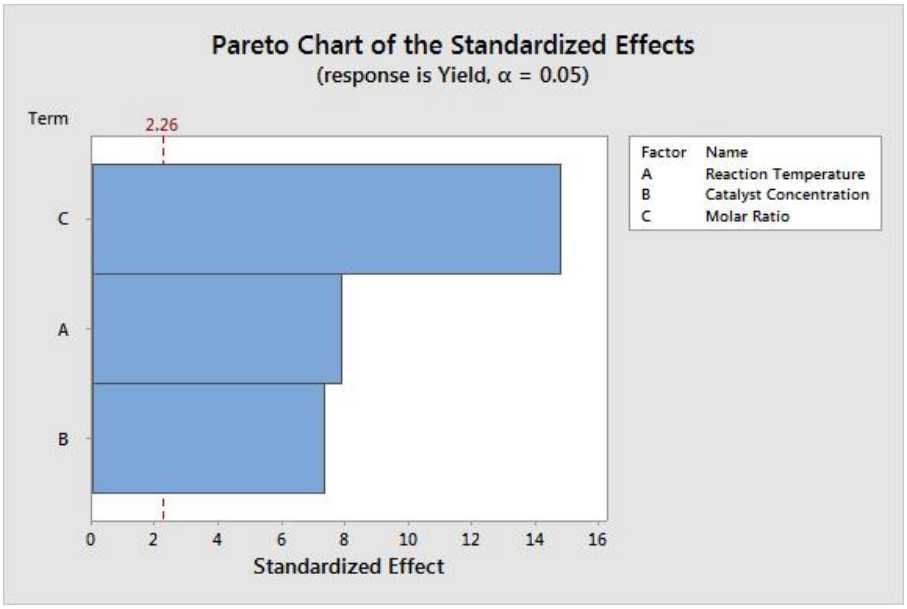

Figure 3. Pareto Chart

3 Result and Discussions

3.1 Physicochemical properties

Physicochemical properties of the biodiesel are gaining more importance, due to the fact, its increasing demand in worldwide. The properties of the biodiesel depend on the chemical compositions and fatty acid compositions. In this paper different physicochemical properties were measured by following international standards. Table 7 represents the different properties and their standards. Fatty acid compositions (FAC) in the biodiesel are the important properties which reveal the information of combustion propensity and exhaust emissions. In this research FAC were analyzed by gas chromatography. Table 7 represents the contribution of saturated and unsaturated FAC in the biodiesel. From the researchers (Kolakoti and Rao, 2017b) it is found that, the presence of unsaturated FAC level in the biodiesel will increase the formation of NOx and unburnt hydrocarbons.

Table 7. Fatty acid composition by Gas chromatography

S.No	Composition	Saturated/Unsaturated	Wt%
1	Palmitic acid	Saturated	22
2	Linoleic acid	Unsaturated	52
3	Oleic acid	Unsaturated	7
4	Myristic acid	Saturated	4
5	Caprylic acid	Saturated	3
6	Stearic acid	Saturated	12
			100

The quality of combustion depends on the quality of the fuel, Cetane number (CN) is known for the diesel quality index. In general, the CN for biodiesel is always greater than the conventional diesel fuel. The cost involved in determining the CN through experimental approach is high and sometimes even less accurate due to the experimental errors. In order to overcome the experimental cost and errors different methods have been proposed to predict the CN. Bamgboye and Hansen (2008) suggested that, with the FAC it is possible to predict the CN. Equation (2) represent the relation between biodiesel FAC and CN.

$$\textbf{Cetane number} = K+ax_2+bx_2+cx_3+dx_4+ex_5+fx_6+gx_7+hx_8- \quad (2)$$

(Bamgboye and Hansen (2008)

$$61.1+0.088X_2+0.133X_3+0.152X_4-0.101X_5-0.039X_6-0.243X_7-0.395X_8$$

where K, a, b, c, d, e, f, g, h are constants taken from regression analysis and X_1 to X_8 are the % of FAC

For Waste Fried Palm Oil Methyl Ester (WFPOME),

Cetane number = 61.1 + 0.352 + 2.926 + 1.848 – 0.273 – 12.636 = **53.32**

3.2 Maximum Yield

Biodiesel yield is defined as the ratio of weight of produced biodiesel to weight of raw oil. From the response Table 3, the optimum values were generated using response optimizer in DSD and the optimum values were found as reaction temperature 55, catalytic concentration 5 and the molar ratio as 6 as shown in optimization plot.4 a & b and in the Table.8

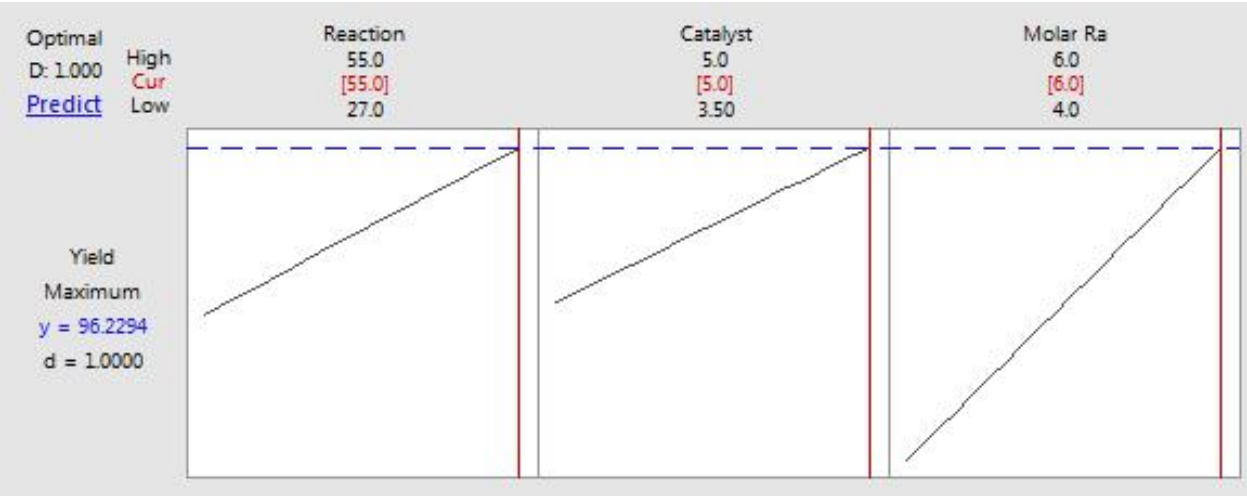

Figure 4(A). Main effects plots for yield.

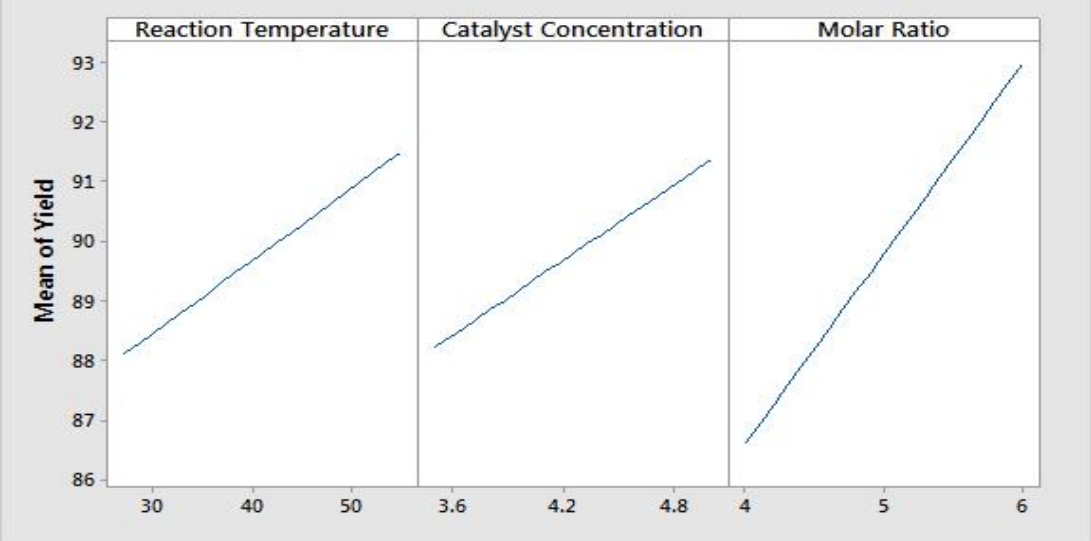

Figure 4 (B). Main effects plots for yield.

Table 8. Optimum contributing factors

S.No	Variable	Setting
1	Reaction Temperature	55
2	Catalytic Concentration	5
3	Molar Ratio	6

Regression equation was generated for the three factors and optimum yield was found out from the regression equation (Eq.2) by substituting the optimum values in the equation a maximum yield of **96.230** was obtained.

$$Yield=60.14 + 0.1206\ R.T + 2.093\ C.C + 3.165\ M.R = 96.230 \quad (2)$$

Where

R. T= Reaction Time

C.C= Catalytic Concentration

M.R= Molar Ratio

3.3 Effect of molar ratio on yield

Molar ratio (methanol to oil) plays a vital role in the transesterification process. The yield of the methyl esters can be increased with increasing molar ratio to an optimum level. This may be due to high mass ratio of reactants, which increases the contact between the methanol and the oil molecules. If the percentage of alcohol (methanol) increases than the desired for a given mixture then the polarity of the mixture will increase there by increasing the solubility of the glycerol back into the ester phase which ultimately reduces the biodiesel yield. From the figures 5&6 of contour and surface plots it is clear that, more than 94% of methyl ester yield can be acclaimed at a molar ratio (M.R) of 6.0, catalytic concentration (C.C) of 5 at a constant temperature (C.T) of 41^0C.

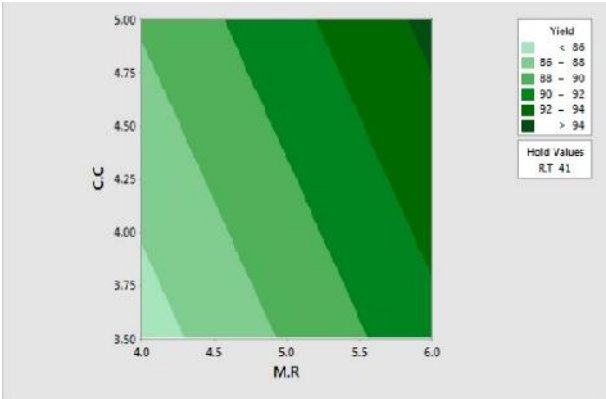

Figure 5. Contour Plot for max yield

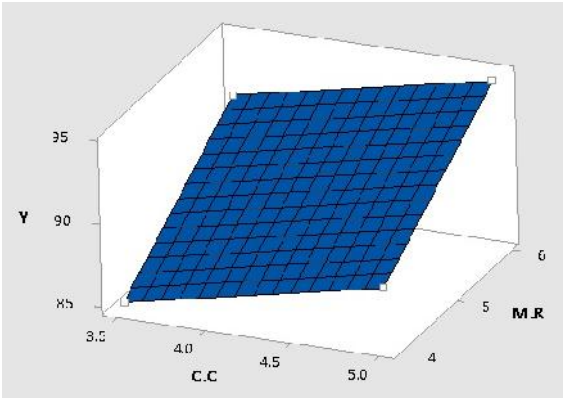

Figure 6. Surface Plot for max yield

3.4 Effect of reaction temperature on yield

To achieve maximum yield, reaction temperature and the presence of alcohol in a given mixture are very important. At high temperature (<62^{0}C) the rate of reaction in the mixture is more and the contributing yield will increase. If the reaction temperature exceeds the boiling point of methanol (64.5^{0}C) then evaporation of methanol takes place there by decreasing the volume of the mixture which will result in decrease in the biodiesel yield. From the figures 7 & 8 a maximum yield of 94% is achieved at a reaction temperature of 55^{0}C, molar ratio of 6 by maintaining a constant catalytic concentration of 4.25.

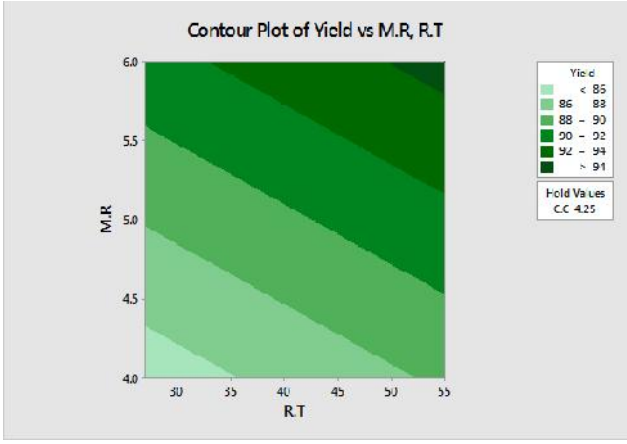

Figure 7. Contour Plot for max yield

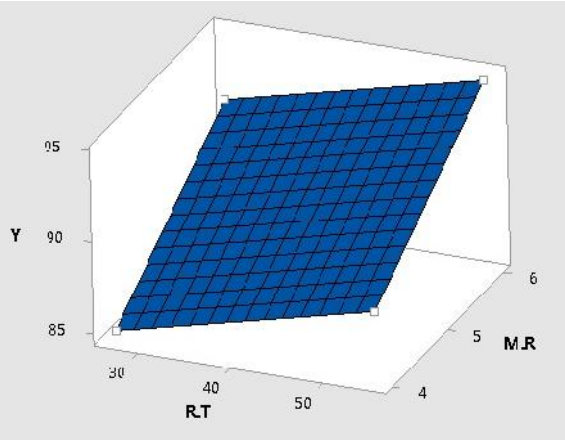

Figure 8. Surface Plot for max yield

3.5 Effect of Catalytic concentration on yield

Different catalysts were used for the production of methyl esters some of them were sodium hydroxide (NaOH) and potassium hydroxide (KOH). Due to the wide availability and for better biodiesel yield sodium hydroxide is used as catalyst. In this present analysis 3.5 to 5 grams of NaOH is used in the transesterification process. Figures 9 & 10 represents the contour and surface plots for the WFPO biodiesel yield at different catalytic concentration and different reaction temperature. From the figures 10 & 11 it is clear that by increasing the CC (4.75 to 5 grams) and the RT (47 to 55^{0}C) simultaneously the maximum yield (93%) of the WFPO biodiesel is achieved by maintaining a constant molar ratio of 5.

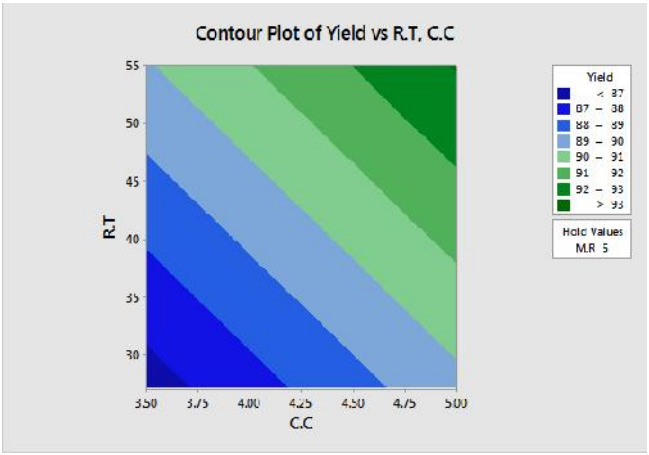

Figure 9. Contour Plot for max yield

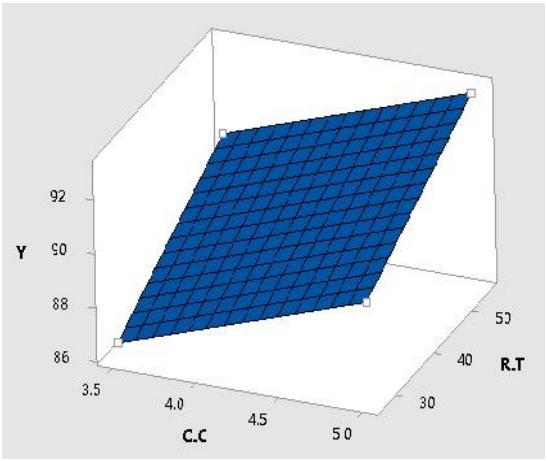

Figure 10. Surface Plot for max yield

3.7 Model Validation

Equation (Eq1) developed by regression analysis represents the theoretical yield. It is validated by conducting the experiments by using the three process parameters of methanol to oil ratio, catalytic concentration and reaction temperature. In order to achieve the accurate response yield, experiments were repeated for three times and the average yield was calculated as 95%. The experimental yield result was in a reasonable agreement with the predicted yield results with an error of 1.230% which may acclaim as human error and can be neglected. Thus, from the experimental results it can be said that the definitive screening optimization is effective in predicting the important response parameters for the biodiesel production.

3.8 Glycerin to soap preparation

The byproduct of transesterification process is the glycerin. In this research an attempt was made to utilize the glycerin by converting it to a soap for cleaning the glass beakers which are used in transesterification process.250ml of solidified glycerin was heated up to70^{0}C to remove the methanol from the glycerin.10grams of NaOH was mixed with 62.5 ml of hot water, this mixture was poured into liquid glycerin and it was allowed to mixed at slow speed. Essential oil (Lavender) was added for fragrance and the mixture was poured in a mold and it was allowed to cool for 8 hours. Figure 11 represents the flow chart of soap making.

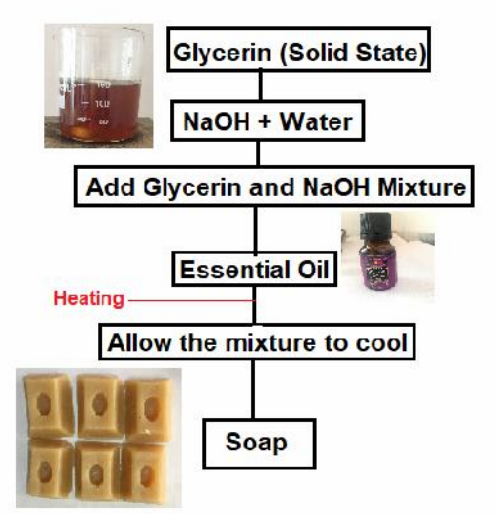

Figure 11. Flow chart of soap production

3.9 Cost analysis

The production cost of the biodiesel is mainly dependent on the raw oil cost and the catalyst which are used in the transesterification process. The present study focusses on the raw oil which is available abundantly after their usage. An internal survey was conducted in the college campus regarding the availability of oil after their usage. It is observed that around 85 liters of oil per day is dumped. Hence, the waste fried palm oil is used for the biodiesel production and the detailed cost involved in the process is shown in the Table 9. The cost of biodiesel is Rs 61.27/- which is lower than the present diesel cost of Rs 70/-.

Table 9. Cost calculations of waste fried palm biodiesel.

S.No	Material Used	Quantity Used	Price/Liter/Kg	Rs
1	Waste fried Palm oil	1 liter	0	0
2	Methanol	260 ml	160	41.6
3	H_2SO_4	2ml	35	0.07
4	NaOH	8 grams	450	3.6
5	Distilled water	1 liter	6	6
6				51.27
7	Miscellaneous cost			10
8	Total Cost of the WFPME			**61.27**

4. Conclusions

In the present analysis, production of biodiesel from waste fried palm oil was investigated and optimized under the influence of three process parameters i.e. molar ratio, catalytic concentration and reaction temperature by definitive screening design. The obtained waste fried palm methyl was characterized to estimate its important fatty acid compositions, physical and chemical properties by following international standards. Based on the experimental results the following conclusions are illustrated.

- The experimental optimum process parameters were determined using definitive screening design were: molar ratio, catalystic concentration and reaction temperature with the corresponding yield of 96.230%
- The molar ratio was observed to be most influencing parameter with a contribution factor of 6:1 followed by reaction temperature of 55^{0}C and the least influence parameter was catalytic concentration.
- The regression model was developed and validated by confirmatory experiments at optimum conditions.

- In order to achieve the accurate response yield, experiments were repeated for three times and the average yield was calculated as 95%. The experimental yield result was in a reasonable agreement with the predicted yield results with an error of 1.230% which may acclaim as human error and can be neglected
- Fatty acid compositions were analyzed by gas chromatography and found that unsaturated fatty acid compositions were 59% followed by saturated fatty acid compositions of 41%.
- The obtained methyl ester was characterized for different physical and chemical properties by following the international standards and the obtained results were closely matches with standard diesel fuel properties.
- The byproduct of transesterification process was glycerin and it was converted to soap.
- The cost analysis of waste fried palm biodiesel was low than the standard diesel fuel cost.

Hence, waste fried palm oil can be considered as most suitable for biodiesel production and it will reduce the initial raw oil cost of the oil during the transesterification process. Advanced optimization technique of definitive screening design was successfully tested and can be implemented.

Nomenclature

CC	Catalytic Concentration
CN	Cetane Number
DOE	Design of experiments
DSD	Definitive screening design
MR	Molar Ratio
RT	Reaction Temperature
WFPME	Waste frying palm methyl ester
WFPO	Waste frying palm oil

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