OPTIMIZATION OF NEEM OIL METHYL ESTER USING RESPONSE SURFACE METHODOLOGY (RSM)

Amol M. Ramning[1], Ganvir V. N.[1], Aditaya Akheramka[1], Y.C. Bhattacharyulu[2]

[1]Department of Petroleum Refining & Petrochemical Technology,
Laxminarayan Institute of Technology, Nagpur, Maharashtra, India.
[2]Department of Post-Graduation in Chemical Engineering,
Anuradha Engineering College, Chikhli, Maharashtra, India.

ABSTRACT

Fast depletion of world's petroleum reserves and increasing ecological concerns has created a great demand for environmentally eco friendly renewable energy resources. Biodiesel has emerged as a sustainable alternative to petroleum origin diesel and its usage have been encouraged by many countries. Transesterification process depends upon a number of process parameters which are required to be optimized in order to maximize Biodiesel yield and minimizing the acid value. Neem Oil is a Non edible vegetable oil, Neem Trees are very commonly found in south-eastern Asia i.e. India and its potential suitability as a biodiesel feedstock is still not evaluated comprehensively. In this research paper, the transesterification process for production of Neem oil methyl ester has been analyzed for getting lower viscosity by use of statics and mathematics which can be optimize the process parameters in very short time by using optimization method. Central Composite Design (CCD) and Response Surface Methodology (RSM) were utilized to determine the best operating condition for both esterification and transterification process. The Optimum condition for Esterification process is 16.832 cSt against 1:9 Alcohol to Oil Molar ratio, 10 (minutes) Reaction Time and 3ml H_2SO_4 Catalyst Concentration.

KEYWORDS: *Biodiesel, Neem Oil, Oscillatory Baffled Reactor, Transesterification, Response Surface Methodology.*

I. INTRODUCTION

Fast depletion of world's petroleum reserves and increasing ecological concerns has created a great demand for environmentally benign renewable energy resources [1].There is an increasing worldwide concern for environmental protection and for the conservation of non renewable natural resources. For this reason the possibility of developing alternative energy source to replace traditional fossil fuels has been receiving a large interest in the last few decades [2].

There are various renewable source of energy successfully tried and used by different nations to limit the use of fossils fuels. Among this renewable sources of energy include, solar energy, wind energy, geothermal energy, tidal energy, ocean thermal energy, hydropower and biomass. Among the renewable sources solar energy, wind energy are not continues and we have to depend upon their availability. The energy conversion methods for these sources are also very costly which increase the price of the fuel. Among the alternate fuels for the petroleum fuel, vegetable oil esters (biodiesel) have gained good promise and suitability for their use in compression ignition engine as a fuel [3].

Biodiesel is considered as a possible alternative and future fuel for diesel engine due to the predicted shortage of fossil fuels and increase in the price of the petroleum. It is biodegradable, non-toxic, and environmentally benign with low emission profiles. Biodiesel, a mixture of alkyl esters of fatty acids, is usually synthesized by the base-catalyzed trans-esterification of oils or fats with short chain alcohols. Different types of catalysts such as base, acid or lipase are used in Transesterification for biodiesel synthesis but the base-catalyzed reaction is the most common in the industry due to easier, faster and cheaper processing [4].

There are number of edible oil available in market i.e. Sunflower oil, Soyabean oil, Cotton seed oil, coconut oil, Ground nut oil etc. from which preparation of biodiesel can be achieved. But use of edible oil as edible oil is generally used for cooking purpose. Similarly if edible oil is used for biodiesel production it can cause the shortage of oil for cooking and the cost of oil may go high [5]. However, it may cause some problems such as the competition with the edible oil market, which increases both the cost of edible oils and biodiesel. In order to overcome these disadvantages, many researchers are interested in non-edible oils which are not suitable for human consumption because of the presence of some toxic components in the oils [6].

Manufacturing of Biodiesel from non edible oil will be a good threat compared to the petroleum fuels. With the abundance of forest and tree-borne non-edible oils to use the esters of these non-edible oils as the alternative fuels for diesel engine. The use of biomass as a source of production of biodiesel as a fuel production is investigated, in terms of its productivity, practicality, and innovative potential to create a cost competitive, environmentally friendly, and renewable source of liquid fuel [7]. The various non edible oil which can be used for the preparation of biodiesel are Jatropha oil(*J. curcas*), karanja oil(*P. pinnata*), Beef tallow oil, used frying oil, other waste oil and fats, Pongamia pinnate oil, Kokum oil, Mahua oil (*M. indica*), Simarouba oil, willed apricot oil , Jojoba oil ,Tobacco seed (*N. tabacum L.*), Rice bran, Mahua Rubber plant (*H. brasiliensis*), Castor, linseed, and Microalgae , Kusum oil, Neem(*A . indica*) and Sal oil. But the use of this oils as a feed stock for production of biodiesel may varies as per there availability in different parts of the world [3].

The Biodiesel can be prepared by use of Neem oil as a feed stock. As Neem trees are commonly found in Southeast Asia i.e. India, A mature Neem tree produces 30 to 50 kg fruit every season. The neem seed has its high oil content of 39.7 to 60%. The neem oil can be available without shortage of feed stock and with lower cost. Neem oil contains a high percentage of monounsaturated fatty acids (C16:1, C18:1), a low proportion of polyunsaturated acids (C18:2, C18:3) and a controlled amount of saturated fatty acids (C16:0, C18:0). The aforementioned characteristics of neem oil plants and its fatty acid composition of the oil make it to be a useful renewable source for biodiesel production [8][9].

The production of a fuel is experimental process which involves the experimental process like the reaction time required for the chemical process, the reaction temperature, the type catalyst used and its concentration, Number of stages required, the reaction time required for the process, the type of reaction method. This all data cannot be received by literature survey. Some experimental works have to be carried out to get a final conclusion. The Experimentation is an application of treatment to experimental units, and then measurement of one or more responses. With the increasing competition and existence in market, the manufacturer needs to produce the products i.e. quality and quantity with a least cost.

This can be achieved with experience and research in the corresponding field but these things require plenty of time. In recent time there is a method which can be used which works on the principle of statics and mathematics which can optimize the process parameter in very less time. This Mathematical modelling of the process is called as **RESPONSE SURFACE METHODOLOGY (RSM)**. It is a scientific method which require observed and gathered data regarding the working of process and system works with less use of chemicals, laboratory equipments and setups, manpower, and saves valuable time with getting perfect results [10].

Response surface methodology is a collection of mathematical and statistical technique which are useful for the modelling and analysis of problems in which a response is influenced by several variables. The most extensive application of RSM can be found in the industrial environment in the situation where a number of input variables affect some performance measures called the response in ways that are not easy or unfeasible to depict with a rigorous mathematical formulation [11].

In the present study we have tried to analyze the reduction of viscosity of biodiesel in a two stage process. The most important property of a fuel is its viscosity. Low viscosity of the oil is the ideal parameter for performance of the engine. High viscosity of the fuel causes engine problems like severe engine deposits, injector choking, piston ring sticking and difficulty in starting of engine i.e. motor vehicle especially in cold weather. Therefore viscosity of the fuel should be less for engine performance point of view and lubrication property [12].

The main factors affecting the transterification reaction are the amount of alcohol and catalyst; reaction temperature, Pressure and reaction time; the content of free fatty acids (FFA) and water in oils. The conversion is very complicated if the Oil contains higher amount of FFA i.e. more than 1%

w/w. Due to high acid value of neem oil a two stage esterification cum transterification process has to be followed to convert neem oil into Neem oil methyl ester (Biodiesel). If 2 stages are not followed the oil sample will get converted in to soap form. This soap can prevent separation of the biodiesel from the glycerine fraction [13].

II. MATERIALS AND METHODS

2.1. Materials

The Neem oil used is obtained from neem seeds. The oil in this present study was obtained from Neem Foundation, Nagpur, India. All the chemicals used were of analytical reagent grades. Potassium hydroxide (KOH) was used in pellet form. The crude neem oil was unrefined but filtered oil is a dark brown in colour. The acid value of crude Neem oil was determined by acid base titration technique.
The acid value Neem oil is 10.92 mg KOH/gm of oil sample. If the acid value of the oil is less than 1.0 mg KOH/gm of oil sample, there is a single stage transterification process used to convert the free fatty acid (FFA) in the oil to the methyl ester. The acid value of Neem oil is more than 1.0 mg KOH/gm of oil sample so here there is a two stage pre-treatment process followed by transterification process. The FFA were first converted to ester in a pre-treatment process using methanol as a reagent and sulphuric acid as a catalyst, with a volume to volume (v/v) ratio of 1-5 % to reduce the acid value of Neem oil near to 2 mg KOH/ gm. The next process is the transterification where triacylglycerol present in treated oil is converted to methyl ester using methanol as a reagent and KOH as an alkaline catalyst.

2.2 Procedure

2.2.1 Pre- treatment

In the pre treatment process, 100 ml of Neem oil was mixed with methanol and 1% -5% sulphuric acid in a 300 ml oscillatory baffled reactor. The reactor is exposed to room temperature throughout the reaction i.e. at 25-30 ^{o}C. The reaction and mixing was carried out under oscillatory baffled agitation in the reactor. Then, the mixture was allowed to settle in a conical bottom separating funnel. After a period of 10 hours the mixture get settled in two layer with the top layer contains fatty acid methyl ester (FAME) and untreated glycerides which were subjected to second step in a transterification process. The bottom layer contains the water and fatty acids were separated from the upper layer [14].

2.2.2 Transterification

The transterification process was carried out in a 300 ml oscillatory baffled reactor. A solid catalyst i.e. KOH was mixed in methanol with 80 ml of treated oil was added in the reactor. The reaction was carried out at room temperature under continues oscillatory baffled agitation. The mixture was allowed to settle in a conical bottom separating funnel. After 10 hours the separation was achieved in a two layer, where the upper layer is the FAME (Biodiesel) separated from the bottom layer i.e. glycerol [14].

2.2.3 Analysis

The composition of methyl ester in the Pre Treatment and transterification was analyzed by using gas chromatography by using GCMS-MS Test method. The capillary column is TR Wax MS (30m × 0.25mm × 0.25um) and helium was used as carrier gas. The operating oven temperature is 280 ^{o}C. The other properties checked were the acid value, density, viscosity in laboratory and calorific value by using IS: 1350(Part 2): 1970. Test method. The optimal condition for the pre-treatment step was analyzed by using LAB FIT software and Design Expert 8.0 Software.

III. RESULT AND DISCUSSION

3.1 Composition of NOME

A mature Neem tree produces 30 to 50 kg fruit every year. It contains a high percentage of monounsaturated fatty acids (C16:1, C18:1), a low proportion of polyunsaturated acids (C18:2, C18:3) and a controlled amount of saturated fatty acids (C16:0, C18:0). The aforementioned

characteristics of Neem oil plants and its fatty acid composition of oil is shown in below table no 1. This shows that Neem oil can be useful renewable source for biodiesel production [3].

Table 1: Fatty acid composition of Neem oil [3].

Fatty acid	Formula	Systematic name	Structure	Wt(%)
Palmitic	$C_{16}H_{32}O_2$	Hexadecanoic	16:0	18.1
Stearic	$C_{18}H_{36}O_2$	Octadecanoic	18:0	18.1
Oleic	$C_{18}H_{34}O_2$	cis-9-Octadecenoic	18:1	44.5
Linoleic	$C_{18}H_{32}O_2$	cis-9,cis-12-Octadecedianoic	18:2	18.3
Linolenic	$C_{18}H_{30}O_2$	cis-6,cis-9,cis-12- Octadecatrienoic	18:3	0.2
Arachidic	$C_{20}H_{40}O_2$	Eicosanoic	20:0	0.8

3.2 Optimization process of pre-treatment step

The optimization of a chemical process i.e. biodiesel using Response Surface Methodology follows the statistical approach which involves three major steps. They are selection of designing experiment, estimation of coefficient based on mathematical model and response prediction along with conformation of mathematical model adequacy [15].

3.2.1 Experimental design

First of all we are working on software LAB FIT for finding the LINEAR, CROSS and SQUARE VALUE for Natural variables. Now we will proceed for finding the value of Viscosity on the basis of linear equation. Firstly select the number of independent variable i.e. Reaction Time (X1), Catalyst Concentration(X2) and Molar Ratio (X3). Put the value of independent variable when software provides the data sheet. Next put the value of Y i.e. Viscosity. The value of X1, X2, X3 and Y is saved by the software. Now we will select the toolbar of curve fitting in the software and develop the equation for Linear Equation as below. This all value are Developed by RSM software by using the independent equation for finding the value of Y i.e. Viscosity for all Runs. i.e. 30 number Runs.

Table 2: Pre-treatment stage optimization of Neem oil

Neem Oil Methyl Ester (NOME) 1St Stage										
Natural Variable					Coded Variable					
	Reaction Time (Sec)	Catalyst Concentration (ml)	Molar Ratio (Ratio)	Viscosity (cSt)	Reaction Time (Sec)	Catalyst Concentration (ml)	Molar Ratio (Ratio)			
Run No.	x1	x2	x3	Y	X1	X2	X3	Linear	Cross	Square
1	10	1	6	18.5	0	-1	-1	17.95861	17.99966	18.1239
2	10	2	6	18.31	0	-0.5	-1	17.81378	17.83433	17.93894
3	10	3	6	18.15	0	0	-1	17.66894	17.669	17.76708
4	10	4	6	18.05	0	0.5	-1	17.52411	17.50366	17.60831
5	10	5	6	18	0	1	-1	17.37928	17.33833	17.46264
6	10	1	6	17.85	0	-1	-1	17.95861	17.99966	18.1239
7	10	2	6	17.5	0	-0.5	-1	17.81378	17.83433	17.93894
10	10	3	6	17.25	0	0	-1	17.66894	17.669	17.76708
9	10	4	6	17.12	0	0.5	-1	17.52411	17.50366	17.60831

10	10	5	6	17.08	0	1	-1	17.37928	17.33833	17.46264
11	10	1	9	16.91	0	-1	0	16.79961	16.79966	16.58801
12	10	2	9	16.84	0	-0.5	0	16.65478	16.65483	16.42355
13	10	3	9	16.61	0	0	0	16.50994	16.51	16.27219
14	10	4	9	16.42	0	0.5	0	16.36511	16.36516	16.13392
15	10	5	9	16.29	0	1	0	16.22028	16.22033	16.00875
16	10	1	9	16.2	0	-1	0	16.79961	16.79966	16.58801
17	10	2	9	16.13	0	-0.5	0	16.65478	16.65483	16.42355
18	10	3	9	16.04	0	0	0	16.50994	16.51	16.27219
19	10	4	9	15.8	0	0.5	0	16.36511	16.36516	16.13392
20	10	5	9	15.62	0	1	0	16.22028	16.22033	16.00875
21	10	1	12	15.85	0	-1	1	15.64061	15.59967	15.72412
22	10	2	12	15.72	0	-0.5	1	15.49578	15.47533	15.58016
23	10	3	12	15.61	0	0	1	15.35095	15.351	15.4493
24	10	4	12	15.49	0	0.5	1	15.20611	15.22667	15.33153
25	10	5	12	15.37	0	1	1	15.06128	15.10233	15.22686
26	10	1	12	15.55	0	-1	1	15.64061	15.59967	15.72412
27	10	2	12	15.41	0	-0.5	1	15.49578	15.47533	15.58016
28	10	3	12	15.36	0	0	1	15.35095	15.351	15.4493
29	10	4	12	15.2	0	0.5	1	15.20611	15.22667	15.33153
30	10	5	12	15.07	0	1	1	15.06128	15.10233	15.22686

Further we take the square equation model to find the best 20 Runs in Design Expert 7 software. The best 20 runs are the developed by the software which provide the same optimum condition and 3D graph which is equivalent to the same condition and graph which can be obtained by using the 30 number of practical experimental runs. We have to provide the data, Temperature (X1), Time (X2) and Catalyst Con. (X3). Next software predict the value of Y from the equation i.e. Viscosity. The software will develop the best 20 Runs suitable for the above parameters. The best 20 runs final sheet provided by the software is shown in below table no 3.

Table 3: Pre-treatment stage best 20 Runs

Run No	Rxn Time(x1)	Coded (X1)	Cat. Con.(x2)	Coded (X2)	Molar Ratio(x3)	Coded (X3)	Viscosity(Y)
1	5	-0.5	5	0.5	12	0.5	3.231435
2	5	-0.5	5	0.5	6	-0.5	4.930962
3	10	0	3	0	9	0	16.27103
4	15	0.5	5	0.5	6	-0.5	36.42418
5	10	0	3	0	3.954622	-1	19.17109
6	10	0	6.363586	1	9	0	15.85801
7	15	0.5	1	-0.5	6	-0.5	36.61024
8	10	0	3	0	9	0	16.27103
9	5	-0.5	1	-0.5	12	0.5	4.203894
10	5	-0.5	1	-0.5	6	-0.5	6.06742
11	10	0	3	0	9	0	16.27103
12	18.40896	1	3	0	9	0	50.99764
13	10	0	3	0	9	0	16.27103

14	15	0.5	1	-0.5	12	0.5	33.67253
15	10	0	-0.36359	-1	9	0	16.8322
16	1.591036	-1	3	0	9	0	-0.26497
17	10	0	3	0	9	0	16.27103
18	10	0	3	0	9	0	16.27103
19	10	0	3	0	14.04538	1	15.27166
20	15	0.5	5	0.5	12	0.5	33.65048

3.2.2 Statistical analysis

In the mathematical analysis, Quadratic models were established by using methods of least square. Firstly we will develop the linear equation by using the curve fitting software i.e LAB FIT. The predicted pre- treatment product viscosity is shown in eq. 1

$$Y_{viscosity} = 5.58574 + 1.48357*X1 - 0.144833*X2 - 0.386333*X3. \quad (1)$$

Where $R^2 = 0.87756$.

Here, $Y_{viscosity}$ stands for predicted pretreatment product viscosity value and X1, X2, X3 are the coded values for the factor value (Reaction Time, Catalyst concentration and Methanol- oil ratio, respectively).

We will repeat the above procedure to find the value of Viscosity (Y) using Cross equation.

$$Y_{viscosity} = 9.41739 - 1.118860*X1 - 0.988760*X2 - 1.089045*X3 - 0.0782427*X1*X2 - 0.06822124*X1*X3 + 0.0068333*X2*X3. \quad (2)$$

Where $R^2 = 0.878084$.

Similarly, we will repeat the above procedure to find the value of Viscosity (Y) using Square equation.

$$Y_{viscosity} = 4.9341 - 0.565494*X1 - 0.483244*X2 - 0.899795*X3 - 0.0237625*X1*X2 - 0.01790364*X1*X3 + 0.00683331*X2*X3 + 0.12862740*X1\text{^}2 - 0.00654761*X2\text{^}2 - 0.0373332*X3\text{^}2. \quad (3)$$

Where $R^2 = 0.901681$.

Here we will select the equation among linear, cross and square whose value of R^2 is near to 1. Here the value of Regression correlation R^2 for square equation is 0.901681 is nearer to 1.0 compare to cross and linear equation value.

Now we will use the software Design Expert 7.0 to analysis the variance in order to check that the experimental data is adequate and fits the mathematical model. Figure 1 compares the observed experimental pre- treatment product viscosity with predicted pre- treatment product viscosity. The R^2 value for the pre- treatment product viscosity is 0.901681 i.e.90.1681% of the variability in the data is accounted to the model. The empirical model is adequate to explain most of the variability in the assay reading which shows that it should be at least or above 0.75[4].

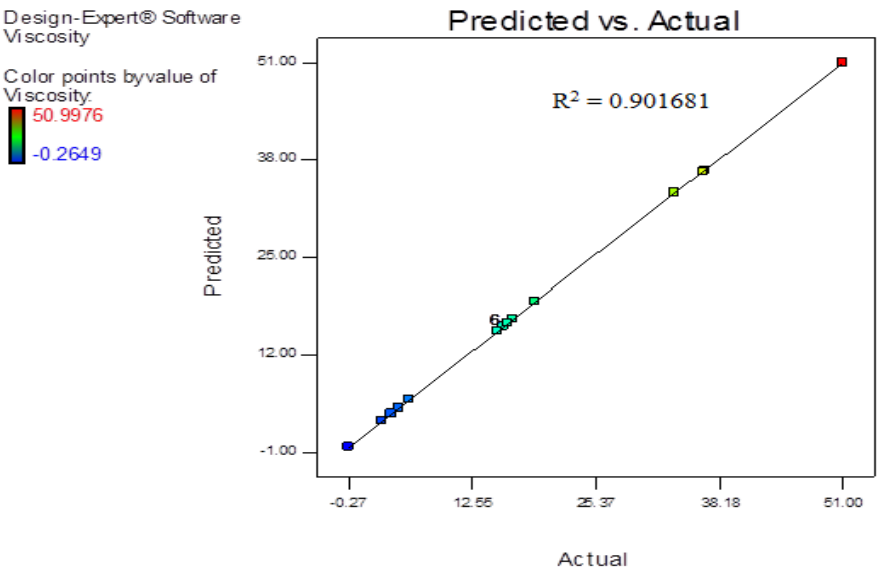

Figure 1: Pairing plot for observed and predicted pre- treatment product viscosity

This indicates that the empirical model gives good prediction on pre- treatment product viscosity at high confidence level of 91%.

3.2.3 Relationship of manipulated variable

The empirical model has been plotted in a 3D surface which represents the response i.e. pre- treatment product viscosity as factor of function of two experimental variables (Fig. 2-4). The relationship between reaction time and catalyst concentration at molar ratio 1:09 is shown in Figure 2.
The Figure 2 show that for high molar ratio pre- treatment product viscosity increases with increasing amount of methanol. The pre-treatment product viscosity decrease when the reaction time is less and viscosity increases with increase in reaction time. Similarly, with increase in catalyst concentration the viscosity of the pre treatment product increases and with less catalyst concentration the viscosity decreases.

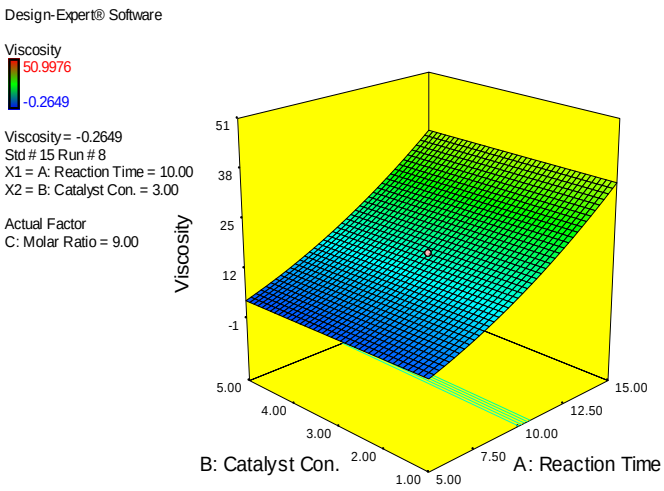

Figure 2: 3 D Graph for Catalyst Conc. Vs Reaction Time for Viscosity

Figure 3 shows the response surface plot of viscosity as a function of reaction time and molar ratio. As the reaction time increases the viscosity of pre-treated product increases and with less time the viscosity also decreases. Similarly the response plot shows that as the molar ratio does on increasing the viscosity of pre-treated oil decreases.

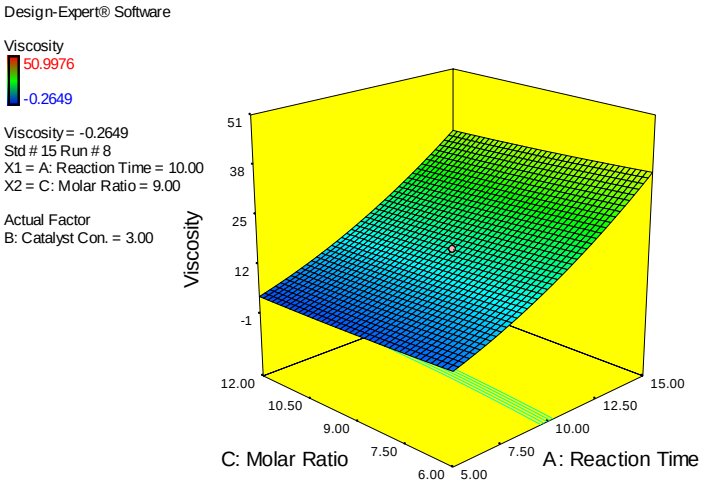

Figure 3: 3D Graph for Molar Ratio vs. Reaction Time for Viscosity

Figure 4 shows the various effects of catalyst concentration and molar ratio on the viscosity of pre-treatment product. As the molar ratio increases the viscosity of the pre-treated product increases. Similarly as the catalyst concentration goes on increasing the viscosity of the pre-treated product goes on decreasing.

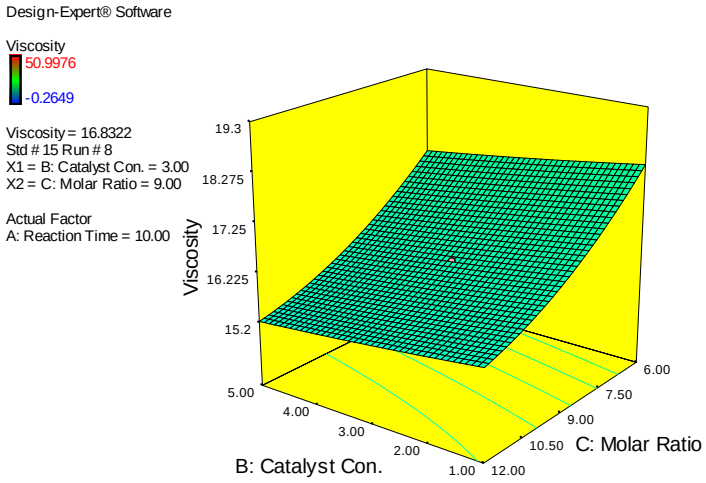

Figure 4: 3D Graph for Catalyst Conc. Vs Molar Ratio for Viscosity

As the molar ratio to catalyst concentration against viscosity interaction is positive. According to the model equation, the molar ratio and catalyst concentration are the most significant factor in esterification process.

3.2.4 Optimization analysis

The response surface analysis using Design Expert 7.0 software indicated that the optimum condition for the predicted condition pre treatment product viscosity is 16.8322 cSt with 1:09 of methanol to oil molar ratio, 3 ml sulphuric acid catalyst concentration and 10 minutes of reaction time.

3.2.5 Transterification step

The pre-treated product produced at the optimum condition in the esterification process went through transterification process in order to decrease the viscosity of NOME (biodiesel). Further the

transterification reaction was carried out using KOH as a catalyst. Methanol was used as a solvent. The reaction was carried out for 10 minutes in an oscillatory baffled reactor, at room temperature. Where viscosity obtained of NOME in transterification stage is 6.448 cSt.
The reaction was further settled overnight at room temperature for complete separation. The GC-MS analysis indicates the conversion of FFA into methyl ester i.e. Palmatic acid methyl ester and Tetradecanoic acid methyl ester. Further glycerol was easily separated from FAME since both components are insoluble in solvent and oil.

IV. FUTURE WORK

The work can be further extended to innumerable situations in industrial processes for estimation of multiple parameters in production of Biodiesel. The ester of this oil can be used as environment friendly alternative fuel for diesel engine creating a greener environment in the future. Its optimization can be easily carried out with the use of optimization tool Response surface Methodology (RSM).

V. CONCLUSIONS

The Response surface methodology and central composite design optimization tool is effective to determine the optimum condition for the process and the interconnected relationship among reaction time, molar ratio of methanol to oil and catalyst concentration with respect to lowered viscosity of treated oil in the pre- treatment step. The optimum condition for pre- treatment step was estimated to be 16.832 cSt Viscosity of NOME pre-treated, 1:9 molar ratio of oil to methanol, 10 minutes for reaction time, 3% H_2SO_4 at catalyst concentration.

ACKNOWLEDGEMENTS

The authors will like to express their sincere gratitude to Dr. S. D. Garway, Director - Anacon Labs, Nagpur for their analytical help for Analysis of composition in biodiesel by gas chromatography and calorific value test. The authors would like to express their sincere gratitude to Dr. S. D. Dawande, Director- Laxminarayan Institute of Technology for allowing use to conduct the practical work in the Petrochemical laboratory, Laxminarayan Institute of Technology, Nagpur.

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AUTHORS

Y. C. Bhattacharyulu is a Senior Professor at Department of Post Graduate in Chemical Engineering at Anuradha Engineering College, Chikhli.

Vinod Ganvir is working as an Assistant professor at Department of Petroleum Refining and Petrochemical Technology at Laxminarayan Institute of Technology (LIT), Nagpur since 2003 to till date. He is a M. Tech. Chemical Technology. He has worked as a lecturer at Chemical Engg. Department at B. A. T. U. Lonare in 2002-2003.

Amol M. Ramning is a M. Tech Chemical Technology from Laxminarayan Institute of Technology, Nagpur. He has worked at S. H. Chem. Tech., Pune as a Marketing Engineer. He has worked as a Project Engineer at Mojj Engineering system Ltd, Pune.

Aditaya Akheramka is a B. Tech. Petroleum Refining and Petrochemical Technology from Laxminarayan Institute of Technology, Nagpur.