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Investigation of Biodiesel Production from High Free Fatty Acid through RSM

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Abstract

Background/Objectives: Diesel engine's optional fuel known as Biodiesel holds fatty acids alkyl monoesters from oils of vegetable or fats of animals. It can be formed from renewable sources namely vegetable oils, restaurant waste oil and fry oil. Bio Diesel may be cost effective if produced from feedstock of low cast namely restaurant animal fats, waste oil, and fry oil, which contains of free fatty acids having high amount (FFA). **Methods/Statistical Analysis:** When processing these oils that are low cost problem occurs and fats are those they regularly possess huge quantity of Free Fatty Acids (FFA) which is impossible for conversion as biodiesel by means of an alkaline catalyst. In this work, a technique has been described for reducing the free fatty acids content of this feedstock's utilizing pretreatment of an acid catalyzed to esterify the free fatty acids earlier to transterifying the triglycerides with catalyst of an alkaline to fulfill the reaction. Chief principle of this work was to expand a two-step production technique of biodiesel from pork waste as a raw material. The variables were methanol to oil ration, base catalyst and acid concentration. With particular attention for optimizing, the first step was the acid catalyst esterification to reduce the free fatty acid content and the second step was alkali catalyzed Transeseterification to convert fatty acid methyl ester. Experiments established the RSM model validity. Maximum percentage of fatty acid methyl ester under optimum conditions of the variables was 93%. **Findings:** Optimum condition for Transeseterification was 13:1 of methanol to oil, 0.4gm sodium hydroxide concentration and 90min of reaction time. Optimum condition for the acid catalyzed esterification was found to be 1.5v/v. ANOVA analysis has been executed for studying the effect of the variables and response surfaces were plotted. Experiments established the RSM model validity. **Applications/Improvements:** Experiments are going to be establishing the RSM model validity along with tuning with the help of intelligent algorithms.

Keywords: Adaptive Steganography, Enhanced Canny Operator, Ensemble Classifier, Least Significant Bit, Positive Predictive Rate

1. Introduction

On rising pattern of modernization and industrialization, the world energy requisite is increasing day by day. Due to their exploration, the petroleum fuels sustained as chief source of conventional energy. Simultaneously, reserve is also identified to be reserve. Both the factors contributed to a pointed rise in prices of petroleum. Hence, petroleum

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fuels are at present the leading source of globe that is CO_2 emissions and their combustion is posing stronger threat for cleaning environment. Petroleum prices hike sharper and rise in environmental pollution together necessitated exploring a few exchange to petroleum fuels that are conventional. In the midst of the other probable options of the liquid fuels a range of vegetable and animal oils have been preferred as suitable option due to prevalent fuel properties[1,2]. Biodiesel production is a highly modern and researcher's technological area because of the significance which is wining daily due to the rise in the price of petroleum and benefits of environment. Regular widespread way to produce bio diesel is the animal facts and Transeseterification of vegetable oils. Transeseterification is an old process and has been conducted as early as 1853 by two scientists J. Patrick and E. Duffy. From that moment numerous studies were performed utilizing various oils namely waste cooking, cotton seed rapeseed, sunflower seed, soybean, frying, winter rape dissimilar alcohols namely methanol, ethanol, butane together with dissimilar catalysts, homogeneous ones namely sodium hydroxide, sulfuric acid and supercritical fluids or enzymes such as lipases, potassium hydroxide. Biodiesel pulls out concentration world wide as an optional fuel for automotive because of petroleum products depletion at earlier rate and regulations of strict environment. Biodiesel is a sulfur free, non-toxic, oxygenated, biodegradable and environmentally friendly optional fuel of automotive and could be produced from sources of renewable namely animal fats, restaurant waste oil, vegetable oils and frying oil and usage of that does not demand any chief modifications in the accessible diesel engine. Chief constraint in high speed usage of biodiesel is the cost of production. Preferable research work, were performed for reducing the biodiesel cost by means of feed stocks at low cost like restaurant waste oil, frying oil and animal fats. These feed stocks at low cost are much challenging for processing since they hold elevated quantity of free fatty acids and the low temperature properties to be poor. Animal fats possess a predominant content of saturated fatty acids and significant properties namely Pour Point (PP), Cloud Point (CP) and Cold Filter Plugging Point (CFPP) of these biodiesels are typically over reasonable limits and typical specifications. Biodiesel from animal fats blending with biodiesel from vegetable oils is an idea of improving these properties with the benefits of iodine value of low range. While methanol is utilized in alcohols is, the reaction is named Methanolysis, where fatty acid methyl ester and glycerol have been produced. Fatty acid methyl esters could be utilized as an optional fuel for diesel engine (biodiesel). Biodiesel is alternate or traditional petroleum diesel extender which could finds its application in conventional diesel engines, and biodiesel usage is beneficial to reduce CO_2, CO, SO_2 and particle materials emission. Worldwide spread of Biodiesel is happening recently. For example, in France, every diesel fuel marketed possesses biodiesel of 5%.

Transeseterification utilizes a conventional alkali catalyzed process provides elevated conversion levels of triglycerides to their equivalent methyl esters in little time. Transeseterification reaction demands a catalyst in the idea of obtaining sensible rates of conversion. Catalyst nature is primary as it brings up the limits of composition which the feedstock has to conform. In addition the conditions of reaction and post partition instructions are programmed by means of the catalyst nature which is involved. At this time the majority biodiesel has been made ready utilizing catalysts of alkaline namely potassium and sodium meth oxides and hydroxides. Technologically, KOH and NaOH were chosen because of their extensive accessibility and economical cost.

Although Transeseterification is possible utilizing base catalysts which are homogeneous, the entire process of base-catalyze undergoes stern restrictions which translate into elevated cost of production for biodiesel. Severe feedstock stipulations are remarkable matter in this process. In specific, the total content of FFA correlated with the lipid feedstock should not go beyond 0.5 wt%. If not, formation of soap critically holds back the biodiesel of fuel grade generation. In the feedstock, formation of Soap occurs on reaction of metal hydroxide catalyst with FFAs. Production of soap contributes to the gel formation, rising viscosity and shoots up the cost of product separation. Alcohol and catalyst too have to comply with exact conditions. Alcohol together with the catalyst have to be necessarily anhydrous (total water content should be 0.1-0.3 wt% or less, which is demanded due to the assumption that in the feedstock, water occurrence contributes alkyl esters hydrolysis to FFAs and, as a result, soap gets formed[3].

Requiring specifications of feedstock for base catalyzed reactions ended up researchers for seeking catalytic and processing choices which could simplify this intricacy and reduces the cost of production.

Methodologies depend on acid-catalyzed reactions holds potential of achieving this because acid catalysts do not exhibit susceptibility which is measurable to FFAs. Due to this cause and also that only some previews exists in biodiesel synthesis area which chiefly undertake the problem of reactions which are acid-catalyzed, progress of acid-catalyzed methodologies is the focal point of this research.

Although fats of animal and waste greases comprise low-cost feedstock sources for biodiesel, their elevated FFAs concentrations create them unsuitable for the conventional straight base catalyzed Transeseterification route to biodiesel because of the concentrations formation of soap. Moreover, as a choice multistep process permits the feed stocks usage possessing FFA concentrations very greater by primarily implementing the acid catalyzed Presterification of the FFAs earlier to the base catalyzed TG Transeseterification. Association of acid catalyzed FFA Presterification subsequent to base-catalyzed TG Transeseterification is normally named the integrated process. In spite of the additional production cost, the process of integration is being gradually more applied to biodiesel production from economical range but greater FFA feed stocks with first-class results. Biodiesel characteristics exists which are a straight outcome of the alkyl chains which are long, linked with the alkyl esters which comprise the bio-fuel and highly rely on the feed stocks utilized. For instance, relying on the composition of feedstock, biodiesel and its blends in cold climates could experience predominantly from high pour points, cold filter plugging points and cloud points. In specific, these issues are highly obvious that biodiesel feedstock's sources are fats of animal. At this time, additional saturated character of the fatty acid composition of fats of animal are transferred to the alkyl esters (biodiesel) acquired. In distinction on preparation of biodiesel from vegetable oils, enclose oleic and linoleic acids esters which possess alkyl chains that are unsaturated. In common, un-saturation degree of the alkyl chains associates much with the cold flow biodiesel performance which refers that the additional double bonds on the alkyl chain, reduced melting point of the esters acquired and hence colder it could get without displaying unwanted high viscosity or solidifying. Issues delineated above could highly restrict the usage of biodiesel organized from fats of animal. Moreover, usage of additives for improving properties of flow of biodiesel at reduced temperatures was proposed as a contradict measure for this issue.

RSM is a group of mathematical and statistical techniques helpful to model and analyze issues by the way of a response towards interest has been affected by numerous variables and the purpose is optimization of this response. In common all RSM issues utilize either one or the combination of the both these models. In every model, the each factor levels are independent of the levels of other factors. In the idea of getting the highly efficient outcome in the polynomials approximation, the appropriate experimental design has to be utilized for collecting data. On collection of data, the Method of Least Square has been utilized for estimating the polynomials parameters. Response surface analysis has been implemented by utilizing the surface which gets fits. Response surface designs are varieties of designs to fit response surface. Hence, purpose to study RSM is given by;

- Accepting response surface topography (ridge lines, local minimum, local maximum), and
- Identifying the region of occurrence of optimal response. Purpose is to shift speedily and professionally over a path for acquiring a minimum or maximum response hence optimizing the response.

Numerous designs exist to fit a second order model. Highly familiar design is the Central Composite Design (CCD).

- To find the suitability of use of pork oil as alternative for diesel fuel due to depletion of fossil fuels.
- To study the properties of pig oil and to compare with that of neat diesel.
- To identify the influencing process variables on biodiesel yield by using RSM.
- To optimize production of biodiesel using acidic pretreatment.

2. Literature Review

Constructed and studied a pilot plant to contribute biodiesel from an extensive choice of feed stocks together with those having great amount of free fatty acids[3]. A pilot plant with 190liter batch was constructed to process high free fatty acid which could feedstocks

involving pretreatment that is acid catalyzed subsequent to Transeseterification which is alkaline catalyzed. Pilot plant case studies scale production of biodiesel from soybean oil, brown grease with 40% free fatty acids and yellow grease with 9% free fatty acids have been exhibited. Changeable reaction parameters effect has been discussed and the process of separating and washing has been briefed. Fuel cost estimates utilizing various feedstocks are also contributed.

Carried out investigation on conversion of a mixture of 75% restaurant waste oil and 25% Pig Fat Oil (PFO) into Restaurant Waste Oil Pig Fat Methyl Ester (RTWOPFME)[4]. Different amounts of methanol (25, 30, 35, 40, 45 and 50 % by volume) alkaline catalyst (NaOH) concentrations (0.3, 0.5, 0.7, and 0.9 % by wt.) reaction temperature (55°C, 60°C and 65°C) and reaction time (90, 120 and 150 min) were selected for the Transeseterification process in the idea of optimizing the experimental conditions for maximum biodiesel yield. Amount of H_2SO_4 (1.5ml) was kept constant. Maximum yield (80% by volume) at optimized process parameters such as methanol (50%) NaOH (0.3g), reaction temperature (65°C), reaction time (90min) and H_2SO_4 (1.5ml) was obtained.

Studied effect of Methanolysis and Ethanolysis on animal fat[5]. Taking Pork fats as two separated samples and single beef tallow which is natural have been straight away Transesterified with a best concluding outcome yield: 87.7, 86.7 and for Methanolysis is 86.3%, and for Ethanolysis78.4, 82.6 and 82.7% correspondingly. Content of Methyl ester has been estimated for being greater than 96.5 mass % for every samples organized. Natural C17.0 occurrence in animal fats makes it essential for correcting the method projected in the standard EN14103 (2003). Biodiesel density at 15°C of the produced biodiesels was in the range of 4.5 to 5.16 mm^2/s, also satisfying demands of EN14214 standard. Value of Iodine is greatly lesser than the greater restriction recognized by EN14214 standard beyond Oxidation Stability (OSI) is much reduced than the mandatory limit, 6h, of the standard which could be attributed to the Edgar Lotero feed stocks with lowered[5] synthesized biodiesel via the lipid molecular weight alcohols Transeseterification. At present, alkaline bases were utilized for catalyzing the reaction whereas catalysts demands conditions of anhydrous and feedstocks with Free Fatty Acids (FFAs) at low levels. Low-cost feedstocks possessing greater FFAs levels cannot be straight away utilized along the base catalysts at present engaged. Well-built liquid acid catalysts are very least sensitive to FFAs and could concurrently perform esterification and Transeseterification. Moreover, they seem to be slower and demands temperatures of greater range for reaction. Nevertheless, acid-catalyzed processes might generate biodiesel from feedstocks which is of low-cost, lessening the cost of production. Even though better, when solid acid catalysts tend to alternate liquid acids, the problems due to corrosion and environment can be kept away from and product purification protocols diminishes, predominantly modifying production of biodiesel and lessening cost. This article evaluates few research associated to biodiesel production involving acid catalysts, together with solid acids.

Studied effect of biodiesel production by mixing frying oil which is waste with pork lard[6]. Biodiesel has been shaped by Transeseterification and quality has been estimated by determining numerous parameters as per to EN14214. Lard weight fraction in the mixture keeps changing from 0 to 1 in 0.2 interval. Production of biodiesel contribution too keeps changing from 81.7 to 88.0 (wt%), the least yields gets hold of utilizing frying oil which is waste and only lard as raw materials. Achieved products satisfied much of the estimated quality specifications as per European biodiesel quality standard EN14214. Least purity (96.5wt.%) has been intimately acquired when waste frying oil has been utilized singly and on incorporating 0.2% of lard in the raw material (96.3 wt%); On the other hand, ranging from 93.9 to 96.3 (wt%) being always nearest to the limit. On evaluating effect of composition of mixture in biodiesel quality, establishing a model was possible to be utilized to predict few of the parameters of biodiesel ensuing from frying oil mixtures which are waste, with lard on occasion of usage of numerous dissimilar lard contents.

Gave special attention to optimize alkali- catalyzed Transeseterification to convert Fatty Acid Methyl Ester (FAME)[7]. A molecular weight of 900g/mol with high acid (41.70% and linoieic acid (36.98%) and 2.59 mg KO of acid are present in Jatropha oil. A CCD technique has been put in place to experimental design. 20 experiments were present to involve the three examined variables of methanol to oil molar ratio (0.95-11.50), sodium hydroxide (0.16-1.84% w/w) and reaction time (39.55-140.45.) Design expert program which analyzes statistically

by data for finding appropriate model of % Fatty Acid Methyl Ester (% FAME) as a function of the three investigated variables. A full quadratic model was suggested by the program using RSM with an R_2 and adjusted R_2 of 97 and 94% respectively. The optimum conditions for Transeseterification were a methanol to oil molar ratio of 6.00, 1.00% w/w sodium hydroxide and 90min reaction time. The optimum condition obtained a FAME content of 99.87%.

The resulting Jatropha biodiesel properties satisfied both the ASTMD6751 and EN14214 biodiesel standards. The production technique developed could be further applied in a pilot plant[8]. Jatropha biodiesel produced 99.87% of FAME content. In the validation process, the predicted value from the model was closely aligned to the experimental value. The resulting Jatropha biodiesel properties also satisfied both the ASTMD6751 and EN14214 biodiesel standards. In addition, the major costs in Jatropha biodiesel production were related mainly to raw material cost[9]. The optimized Jatropha biodiesel production using sodium hydroxide as a catalyst could be applied in a Jatropha biodiesel pilot plant. The comprehensive use of Jatropha biodiesel in industrial applications will benefit overall food supplies and will reduce energy problems.

3. Methodology

Following are the components required for biodiesel production. It works on the principle of heat generated from the resistance of the coil it is used to heat the feedstock such that any moisture content present in feedstock gets evaporated. To increase the yield from feedstock microwave was used for extraction. Yield from feed stock increased by 50% using microwave irradiation. Biodiesel Kit, It helps in taking the reaction between the feedstock; methanol and catalyst it consist of a stirrer rotated by the means of motor at the desired speed. Separating funnel, it is a separator funnel, also referred as separation funnel; separating funnel, known to be a laboratory glassware piece finds its usage in extractions of liquid-liquid for separating (partition) the mixture components of a among two immiscible solvent phases of dissimilar densities. Distillation apparatus[10], it works on the principle of heat generated from the resistance of the coil. It is used to remove the excess alcohol present in the biodiesel and for heating the biodiesel to 120°C to ensure that no moisture present in the biodiesel. Clean mixture pig fat oil (100ml) was taken and heated to 120°C for 15min in order to melt all solid fats. This mixture is permitted for getting cooled to a temperature of 50°C and then required amount of methanol (99% purity) is added to the heated mixture of each flask. After stirring for 15min, the solution became murky. To this solution H_2SO_4 is added. Stirring continued for 1hr by maintaining temperature at 50°C. After this, heating was stopped and stirring is continued for another 1h. In the meantime, sodium Methoxide solution was set ready by dissolving different amount of alkaline catalyst in 0.12 1 of methanol per liter of oil. Half of the prepared sodium Methoxide solution is added to this mixture and then heated to the required reaction temperature to 60°C. To this mixture, remaining half of the sodium Methoxide solution is added. Heating and stirring continued at different reaction time. Mixture has been permitted for separating and gets settled overnight at top by gravity settling into liquid of golden color and clear biodiesel and at bottom, the glycerol which is light brown. Subsequent day, draining off glycerol takes place, leaving biodiesel and it was water washed three times. Flow charts for acid catalyzed biodiesel production are shown in Figure 1.

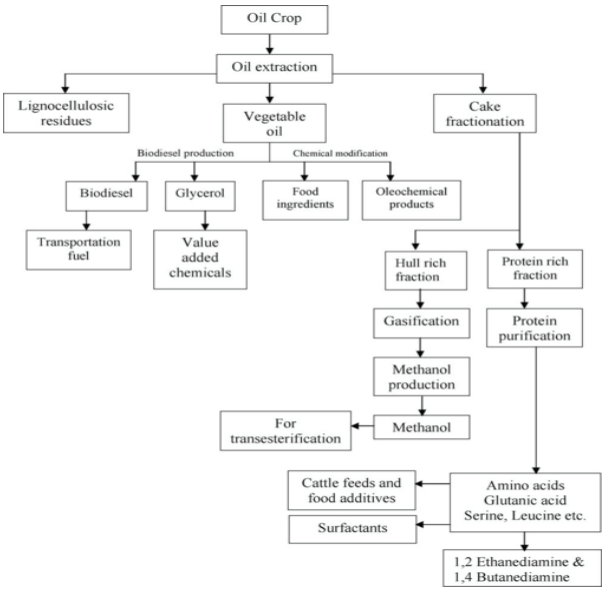

Figure 1. Flow Chart for Acid Catalyzed Biodiesel Production.

4. RSM and Experimental Design

A factorial design of experiments[11]has been implemented for determining the effect of the operational conditions of the synthesis process. Design Expert (Version 8.0.3, Stat Ease, Inc., USA) software has been utilized toper form the graphical analysis and regression of the acquired data. Experimental design implemented in this research was a full 2^3 factorial design possessing around 20 experiments. For evaluating experimental error four central points have been put in additionally and six additional star points are encoded as + α and – α. Experiments were performed randomly for minimizing errors because of possible systematic variable trends. Polynomial model for the yield of lard BD was regressed with respect to the reaction conditions as follows.

RSM is an association of statistical and mathematical techniques[12] which are helpful to model and analyze the issues where an output or response affected by numerous variables and the objective of finding the correlation among the response and variables.

Steps to be followed up in RSM technique are given below:

- Designing of a set of experiments for sufficient and reliable measurement of the true mean response of interest
- Mathematical model determination with most excellent fits.
- Identifying set of experimental factors to an optimum range which gives maximum or minimum value of response and
- On behalf of the direct and interactive process variables effect on the most excellent parameters via graphs that are two dimensional and three dimensional.

When every variable are implicit to be measurable, the response surface could be articulated as follows:

Objective is nothing but optimizing the response variable y and implicit that the independent variables are nonstop and retractable by experiments with trifling errors. Finding an appropriate approximation for the real functional association among variables that are independent and the response surface is required. Regularly a second order model has been implemented in RSM.

$$Y = \beta\ \beta\ \beta_0 + \sum_{i=1}^{k}\beta \sum_{i=1}^{k}\beta_{ii} x_i^2 + \sum_{i=1}^{k}\beta \sum_{i=1}^{k}\beta_{ij} x_i x_j + \varepsilon$$

$$Y = \beta_0 + \sum_{i=1}^{k}\beta_{ii} X_i^2 + \sum_{i=1}^{k}\beta_{ij} x_i x_j + \varepsilon$$

Where random error is denoted as ε, coefficients are referred as β, that has to be found out in the second order model are achieved by the least square method.

Purpose of implementing RSM is not only to examine the response reaches its optimum or optimal value that is closer. On studying cautiously the combination of factors, the response surface model that gives the greatest response could then be presented.

4.1 Developing the Experimental Design Matrix

An experiment, known to be a sequence of tests or trails is where products outcomes that is quantifiable. Due to somewhat great choices of the factors, this has been determined using a level of five, rotatable design matrix, and central composite for optimizing the circumstances of experiment. Central composite rotatable designs of second order were identified as highly competent tool of RSM for associating mathematically with the response surface utilizing negligible probable number of experiments unless bringing down its accuracy. Experimental size is 20 for three independent parameters in the current scenario.

Design has been further divided as three parts as depicted below:

Points constituting a 2^k factorial design, i.e.; 8 points; input variables number is k.

Extra points are added in from a CCD with a. The figure created by these points is named a star. α is value of $2^{k/4}$ in the idea of making the design rotatable. Number of star point is 6 for three independent parameters, and additional 6 points are summed up to centre for giving approximately equal precision for response R_a within a circle of radius 1. Factors those are identified and its limits which are upper and lower limits have been briefed earlier. Units, Notations and their levels selected are illustrated in Table 1.

Table 1. The Notations, Units and their levels chosen are summarized

		Levels				
Process Parameters	Variable	-1.68	-1	0	1	1.68
Oil/Methanol Molar Ratio	A	5	7	9	11	13
Base Catalyst(gm)	B	0.2	0.4	0.8	1.2	1.4
Acid Catalyst(ml)	C	1	1.25	1.5	1.75	2.0

Table 2. Layout of Central Composite Rotatable Design

	Coded Variable			Fatty Acid Ester Content (%)
Trial No.	Oil Methanol Ratio	Base Catalyst (gm)	Acid Catalyst (ml)	Fatty Acid Methyl Ester (%) (FAME)
1	-1	-1	-1	
2	1	-1	-1	
3	-1	1	-1	
4	-1	1	-1	
5	1	-1	1	
6	-1	-1	1	
7	-1	1	1	
8	1	1	1	
9	-1.68	0	0	
10	1.68	0	0	
11	0	1.68	0	-
12	0	1.68	0	
13	0	0	-1.68	
14	0	0	1.68	
15	0	0	0	
16	0	0	0	
17	0	0	0	
18	0	0	0	
19	0	0	0	
20	0	0	0	

Table 2 depicts 20 coded conditions set utilized from the design matrix of central composite which is rotatable comprising of full replication of $2^3 = 8$ factorial design plus 6 centre points and 6 star points. Every selected variables at the intermediary level (0) comprise the centre points and the associations of each of the variables at either it's lowest (-1.68) or highest (+1.68) with the other three variables of the intermediate levels comprise the star points.

Hence 20 experimental tests permitted evaluation of not only linear but also quadratic and offers that are interactive in two way of the variables over the roughness of surface. Cochran WG, Cox, GM briefed the technique of designing central composite design matrix[7]. In recording and processing experimental data conveniently, lower and upper parameters levels are coded to be +1.68 and -1.68. Any intermediary levels coded value could be estimated by utilizing the subsequent expression.

$$x_i = \frac{\left[2X - (X_{max} + Y_{min})\right]}{\left[\frac{X_{max} - X_{min}}{2}\right]}$$

Where upper parameter level is X_{max}, lower parameters level is X_{min} and essential parameter coded values of any value of X from X_{min} to X_{max} is X_i.

5. Results and Discussions

Experimental design matrix and results has been showed in the Table 3 based on central composite rotatable designs.

5.1 Regression Model Equation

Entire design matrixes with experimental values for yield response at the point of design are depicted in Table 3 biodiesel acquired ranged from 64 to 91 %.

Standard experimental matrix for the factorial design and the outcomes of FAME yields are illustrates in Table 3.

Table 3. Experimental Design Matrix

	Coded Variables			Fatty Acid Ester Content (%)
Trial No.	Oil to Methanol Ratio	Base Catalyst (gm)	Acid Catalyst (ml)	Fatty Acid Methyl Ester (%) (FAME)
1	-1	-1	-1	64
2	1	-1	-1	87
3	-1	1	-1	68
4	-1	1	-1	90
5	1	-1	1	68
6	-1	-1	1	88
7	-1	1	1	82
8	1	1	1	70
9	-1.68	0	0	74
10	1.68	0	0	91
11	0	-1.68	0	77
12	0	1.68	0	83
13	0	0	-1.68	71
14	0	0	1.68	87
15	0	0	0	82
16	0	0	0	81
17	0	0	0	82
18	0	0	0	82
19	0	0	0	82
20	0	0	0	81

Here A: Oil to Methanol Ratio, B: Base Catalyst, C: Acid Catalyst concentration.

Analyzing statistically, the regression model has taken placefor evaluatingANOVA. Response surface linear model ANOVA is illustrated in Table 4. Model's p-value was lesser than 0.05 that indicated that the model was appropriate for its usage in this experiment. A lower coefficient value variation (CV = 4.16 %) was obtained. The regression model equation for FAME is as follows;

$$FAME = + 82.34 + 7.72\ xA + 2.04xB + 3.06 \times C - 1.62 \times AxC + 0.38\ xBxC - 0.19\ xA^2 - 0.93 \times B^2 - 1.23\ C^2$$

5.2 Standard Deviation and Coefficient of Variance

Table 4 gives outcomes of standard devotion and coefficient of variance and Table 5 ANOVA) for response surface quadratic model.

Table 4. Standard Devotion and Coefficient of Variance

Standard Deviation	3.36	Root Squared	0.9040
Mean	80.74	Adj R-Squared	0.8176
C.V.%	4.16	Pred R- Squared	0.2634
Press	866.75	Adeq Precision	12.653

5.3 Analysis of ANOVA for Surface Quadratic Model

Model F-value of 10.46 implies the model is significant. Only 0.01 % of chance is that a "Model F-Value" this large might takes place because of the noise. "Prob>F" values less than 0.0500 point out model terms to be significant. In this case A,B, C,AC, BC, A^2, B^2, C^2 are significant model terms whereas AB are insignificant to the response.

5.4 Response Surface Plot

The response surface plot for different the independent variables selected are as follows;

Figures 2, 3 and 4 show the response surface 3D plots indicating effects of interaction between parameters on the yield of biodiesel. Acid pretreatment with lard is an important parameter as it influences the reaction rate of synthesis significantly and subsequent the yield biodiesel. Figure 2 shows the 3D plot of catalyst concentration and acid on the biodiesel yield. The best yield of lard BD could be obtained at 0.8 wt % of base catalyst concentration. From Figure 2 it can be seen that the predicted maximum value of lard BD yield was 91.0 wt %. Hence, the highly appropriate circumstances for the process were obtained by actual trial number 10 molar ratios of 13:1, 1.5 ml acid catalyst and base catalyst concentration of 0.8 wt %. One among the variables influencing the BD yields is the reac-

Table 5. Analysis of Variance (ANOVA) for Response Surface Quadratic Model

Source	Sum of Squares	D_f	Mean Square	F Value	P-Value Prob>F	
Model	1063.77	9	118.20	10.46	<0.0001	Significant
A Oil to Meth	814.28	1	814.28	72.7	<0.0001	Significant
B Base Catalyst	56.92	1	56.92	5.04	0.00486	Significant
C-Acid Catalyst	127.57	1	127.57	11.29	0.0012	Significant
AB	11.52	1	11.52	1.02	0.0051	Significant
AC	21.12	1	21.12	1.87	<0.0001	Significant
BC	1.13	1	1.13	0.100	0.0008	Significant
A2	0.5	1	0.50	0.044	0.0095	Significant
B2	12.42	1	12.42	1.10	0.0063	Significant
C2	21.76	1	21.76	1.93	0.0335	Significant
Residual	112.84	10	11.30			
Lack of Fit	112.84	5	22.57	805.99	0.077	Not Significant
Pure Error	0.14	5	0.028			
Cor. Total	1176.75	19				

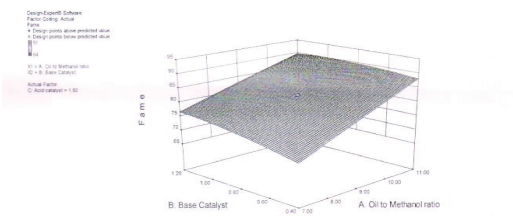

Figure 2. Response Surface Plot for Base Catalyst and Oil to Methanol Ratio.

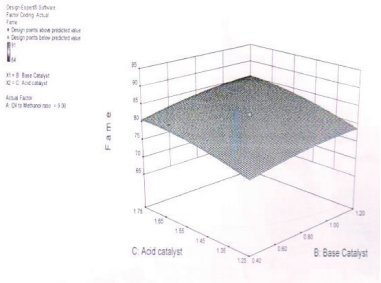

Figure 3. ResponseSurface Plot for Acid Catalyst and Base Catalyst.

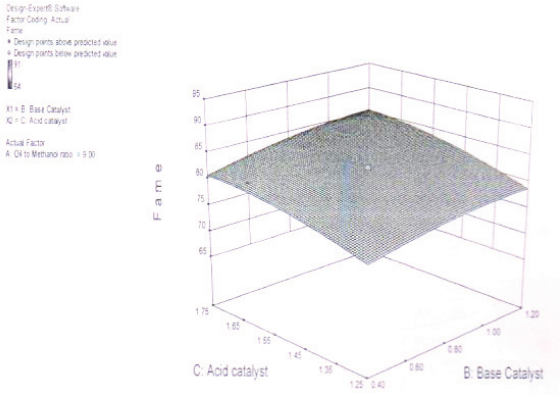

Figure 4. Response Surface Plot for Acid Catalyst and Oil to Methanol Ratio.

tion temperature. Optimized value of temperature was taken from previous research as 60°C. Typically the transesterification reaction temperature has to be less than the alcohol boiling pointin the idea of restricting the evaporation of alcohol. A property was tested and results are shown in Table 6.The constraints taken for optimizations are as follows;

Desirability is a target function which ranges from zero outside the limits tone at the goal. Optimizing numerically identifies a point that makes the most of the desirability function. Characteristic of a goal might be distorted on altering the weigh or significance. For numerous factors and responses, every objective gets associated into single function of desirability are shown in Figure 5. Value is entirely dependent on how intimately the limits which are upper and lower are set associative to the definite optimum. Purpose of optimization is for identifying a first-class conditions set to satisfy every goal, not to reach upto a value of desirability as 1.0. Desirability is just a mathematical technique using RSM for finding optimum values are list in Table 7.

The highest desirable combination was tested experimentally to verify the model. The resulted yield was 93%. Thus the theoretical results were verified experimentally. Properties were tested and results obtained are as follows in Table 8.

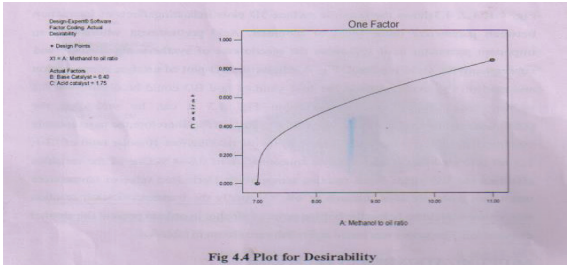

Figure 5. Plot for Desirability.

Table 6. Constraints for Optimization from RSM

Name	Goal	Lower Limit	Upper Limit	Lower Weight	Upper Weight	Importance
A: Oil to Methanol Ratio	Maximize	7	13	1	1	5
B: Base Catalyst	Minimum	0.4	1.2	1	1	3
C: Acid Catalyst	Maximum	1.25	1.75	1	1	4
Fame	Maximum	64	91	1	1	5

Table 7. Solution for Optimization from RSM

Number	Methanol to Oil Ratio	Base Catalyst	Acid Catalyst	Fame	Desirability	
1	13	0.4	1.75	94.678	1	Selected
2	12.98	0.4	1.75	94.608	0.9989	
3	13	0.4	1.75	94.705	0.9988	
4	12.97	0.4	1.75	94.571	0.9984	
5	13	0.41	1.75	94.715	0.9982	
6	13	0.4	1.74	94.764	0.9966	
7	12.93	0.4	1.75	94.445	0.9963	
8	13	0.42	1.75	94.759	0.9961	
9	13	0.43	1.75	94.801	0.9939	
10	13	0.43	1.75	94.822	0.9928	
11	13	0.44	1.75	94.868	0.9903	
12	13	0.4	1.73	94.962	0.9883	
13	13	0.48	1.75	95.035	0.9804	
14	13	0.57	1.75	05.314	0.958	
15	13	0.65	1.7	95.975	0.9147	
16	13	0.82	1.75	95.621	0.8765	

Table 8. Properties of Biodiesel Compared to Diesel

Properties	Pig Fat Biodiesel	Diesel
Kinematic Viscosity (mm^2/s)	4.1	2.46
Flash Point ^oC	161	49
Fire Point ^oC	178	55

6. Conclusion

Two stages Transeseterification processes were used to convert Pig Fat Oil (PFO) into pig fat methyl ester. Transeseterification was carried out with different amounts of methanol alkaline catalyst NaOH concentrations (0.2, 0.4, 0.8, 1.2, 1.6 % by wt.) and fixed reaction temperature 60^{o}C and reaction time 90 min with acidic pretreatment (1, 125, 1.5, 1.75, 2 by volume). From this the optimum quantity of methanol was found out to be 50% by volume. From results we can conclude that;

- High quality biodiesel production is possible from pork fat by using optimization methods. The final product properties fulfilled requirements of biodiesel standards.
- An experiment assures the RSM model validity. The maximum percentage of FAME under the optimum conditions of the variables was 93%. Optimum condition for the alkali catalyzed Transeseterification was 13:1 of methanol to oil, 0.4gm sodium hydroxide concentration and90min of reaction time. Optimum condition for the acid catalyzed etherification was found to be 1.75v/v.
- The molar ratio of methanol to lard is a significant factor affecting the conversion to BD. Stoichiometrically, 3 moles of methanol are necessary for each mole of triglyceride, but in practice a higher molar ratio is engaged in the idea of driving the reaction towards finishing point. In the case of pig fat biodiesel synthesis without pretreatment, yield was increased continuously with rise in the oil to methanol ratio. However, conversion rates were lower than those of pork fat BD synthesis with acid pretreatment. The acid blending raises the reaction rate by preparing the oil soluble in methanol. The highest BD yield of 93.0 wt % has been accomplished when the molar ratio was 13:1 with acid concentration of 1.75ml.
- Optimized value for yield obtained from RSM value.
- In this research, the determination coefficient value (R^2 = 0.9040) depicts that the variation of sample i.e., 90.4% for biodiesel yields is accredited to the variables

that are independent and 9.6% of total variations are not briefed by the model. This could be influenced from other parameters such as temperature, speed of rotation which was taken as constant in this study.

- The most statistically significant factor from ANOVA analysis was oil to methanol ration because the p-value for factor was $p<0.0001$. Acid concentration was also an important factor ($p = 0,012<0.05$). Other factors of the model had no statistically significant effects.
- Least range of the variation coefficient (CV = 4.16) depicts an enhanced precision and the experiments reliability performed model.

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