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# Optimization of Biodiesel from *Citrillus Lantanus* Seed Oil using Alkali-Catalyzed Methanolysis

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***Abstract***— Biomass and agriculture derived materials are alternative sources of energy for the production of biodiesel. Renewable energy attracts wide attention due to its profound contribution to the ecosystem and its role as a sustainable energy source in substitution to diesel and the other petroleum based fuels. This study was conducted to synthesize biodiesel from Citrillus lantanus seed oil. The oil was extracted and characterized having a yield of 49%, density 0.92 g/cm$^3$, acid value 2 mgKOH/g and viscosity of 26 cSt. The oil was characterized to get a high fuel quality in conformity with the American Standard for Biodiesel Testing Materials (ASTM-D6751) and the European Norm (EN-14214) standards. The experiment was designed and analyzed consisting of 28 number of runs by Response Surface Methodology—RSM technique. Four parameters that were studied and varied are temperature and time of reaction, catalyst concentration and methanol-oil ratio, and. The highest triglyceride conversion of 85% was achieved at 55 $^0$C temperature, 30 mins reaction time, 0.5 g NaOH catalyst concentration, and 1:4 methanol-oil ratio. The optimal operating conditions while maximizing desirability was found to be 59.1 $^o$C, 75min, 1.0 g/g, 1:3 of temperature, reaction time, catalyst loading, and methanol-oil molar ratio respectively to give a corresponding yield of 77.13% of biodiesel. The biodiesel produced was in conformity with ASTM standards.

***Keywords—***Biodiesel, Citrillus lantanus, transesterification, optimization, RSM, ANOVA

## I. INTRODUCTION

There is an increasing realization that the global finite reserves of fossil fuels are fast depleting given the fact that global fuel consumption far surpasses the discovery of fresh reserves. It might be recalled that the world had earlier experienced energy crises in the seventies as a result of uncertainty in fossil fuels supply [1]. The transportation sector of Nigeria's economy is perhaps the most susceptible, as it reacts very sensitively to increases in fuel pump price, of which hitherto, a large part is imported into the country. Ironically though, Nigeria is the sixth largest crude oil exporter. This has compelled for the search for sustainable, renewable, and environmentally friendly energy resources [2]. Among the alternatives, ethanol is the most frequently used biofuel as a substitute for gasoline, and biodiesel to substitute for diesel [2]. Biodiesel (fatty acid methyl esters (FAMEs)) synthesized from different vegetable oils, waste oils, and animal fats, may eventually be used to substitute diesel derived from fossil source. Moreover, biodiesel has many prominent properties, notably flash point, cetane number, and volumetric heating value, which are similar to petro-diesel. Biodiesel can be produced via transesterification which removes glycerine from the triglycerides and substitutes it with the alcohol used in the process. This process sustains the heating value and cetane number while significantly reducing the viscosity [3].

## II. RELATED WORK

### Biodiesel Production Techniques

Vegetable oil can be applied directly in diesel engines, but may cause damages like injector chocking, as a result of its high density and viscosity and other unfavorable properties. However, it can be blended with petro-diesel [4]. Biodiesel can be synthesized through various methods such as thermal cracking, micro-emulsion, but the most common method is the transesterification reaction. It is the reaction of an alcohol (like methanol) with the triglycerides that is present in fats, oils, or recycled grease, to produce biodiesel (fatty acid alkyl esters) and the by-product being glycerol. The process is heat intensive and requires a sturdy alkaline catalyst, such as Potassium Hydroxide (KOH) or Sodium Hydroxide (NaOH) [5].

### Biodiesel Characterization

Biodiesel fuel quality primarily depends on the composition of feedstock, mode of production, handling and storage. Fats and oils of divergent origin have different composition of fatty acid [6]. The fatty acid ester content of biodiesel is the same as that of the parent fat or oil it was derived from. Biodiesel feedstock varies with geographical location as well as climate and accessibility [7]. Biodiesel fuel quality is assessed through analysis of physical properties and chemical composition of the fuel. The major problems affecting the quality of biodiesel are contaminants and other minor components due to incomplete reaction i.e., glycerol, alcohol, catalysts, mono-, di-, and triglycerides, and free fatty acid present in the biodiesel. Furthermore, biodiesel composition could be altered due to mishandling and improper storage.

Biodiesel can undergo oxidation during storage and absorb water. Consequently, the effects of these parameters and their engine or analytical test methods are addressed in standards. Biodiesel international standards are imposed in numerous countries. This is in order to make certain that only high grade biodiesel is being commercialized. The most important standards are ASTM D6571 in the United State and EN-14214 (European Committee for Standardization, CEN) in European Union [7].

## III. METHODOLOGY

### Materials and Methods

The materials used in this work are presented from the perspectives of (*a*) raw material used for the experiment (*b*) laboratory setup, apparatus and other equipment used, and (*c*) chemicals and reagents used.

### Raw Material

The major raw material used for the present study was Citrillus lantanus. Fresh fruits were picked from University of Maiduguri woods. The seeds were manually removed from the fruits, cleaned of dirt, sun dried to reduce moisture content, ground to increase surface area. Oil was then extracted from the seeds using a soxhlet apparatus at the Unit Operations Laboratory of the Department of Chemical Engineering, University of Maiduguri.

### Experimental Setup and Equipment

The experimental setup was a batch process and was locally constructed. It comprises of a glass jar with a plastic cover as the reactor vessel. The stirrer was of mechanical form comprising of an electric motor threaded to a metal rod and plastic blades fitted at the end. The stirrer was then braced to a retort stand with a retort clamp, and the blade end submerged into the glass jar. The heating source was an electric water bath. GC-MS QP2010 system and MSD Chemstation was used in characterization of the oil and biodiesel produced. Other equipment used were vacuum oven, jacketed test tube, centrifuge machine, water bath, vortex mixer, weighing balance, density specific gravity meter, bomb calorimeter, seta cloud/pour point refrigerator (D-100), muffle furnace, rotary evaporator, sulphur-in-oil analyser, petro-test viscometer.

### Reagents and Chemicals

The reagents and chemicals used in this work include n-Hexane, methanol, sulphuric acid, hydrochloric acid, sodium sulphate, potassium iodide, sodium hydroxide, potassium dichromate, periodic acid, potassium hydroxide, sodium thiosulphate, soluble starch, glacial acetic acid, linoleic acid, chloroform, aniline, toluene, isopropyl alcohol, phenolphthalein, ethyl alcohol, carbon tetrachloride.

### Methodology

The methodology employed are in respect to (a) preparation of reagents and solutions, and (b) general experimental procedures.

### Preparation of Reagents / Solution

### Oil Extraction

60 grams of ground Citrillus lantanus seed of each sample was placed in a thimble, whose opening was plugged with a piece of clean cotton wool and inserted into a soxhlet extractor that was mounted on a round bottom flask containing 250cm$^3$ n-hexane. A reflux condenser was fitted onto the soxhlet extraction column and the setup was positioned on a heating mantle and heated to 55 $^o$C to vaporize and remove the extraction solvent. The vapour liquefied in the condenser and liquid trickled into the powdered sample that is in the extraction thimble. The solvent dissolves oil content in the powdered sample until the thimble fills up then the solution in thimble was emptied into a round bottom flask. The process continued repeatedly till all oil in the sample was extracted as recommended by [8]. The extraction lasted between five to seven hours depending on sample type after which, the thimble and condenser were dismantled and the oil was recovered from the solvent by heating the flask for 20 minutes on heating mantle at low temperatures (35$^o$). The flask containing the oil was then heated on a water bath to evaporate the remaining solvent out of the oil. The dried oil was transferred into a pre-weighed brown bottle, weighed to determine the yield and kept for subsequent use.

### Biodiesel Production

The oil extracted from the soxhlet apparatus was used for the biodiesel production. The extracted oil was characterized and was found to have a low level of free fatty acids, and will therefore need 1/5th of the weight of oil equivalent of alcohol to treat triglycerides that will result to FAMEs [9].

0.6g of anhydrous NaOH was added to 20.30g (26.0cm$^3$) methanol and stirred continuously till it dissolved. 50g of the oil (triglyceride) was poured in a 250cm$^3$ conical flask and warmed at 40 $^0$C to attain temperature equilibrium. The methanolic NaOH solution was mixed with the oil. The mixture was then stirred with magnetic stirrer on a hotplate at 50 $^o$C for 10, 20, 30, 40, 50, 60 minutes respectively, then the content was then moved into a separating funnel fitted with a filter paper mounted on a retort stand which was allowed overnight to separate under gravity. A dark brown coloured liquid containing glycerol and other impurities was separated from the biodiesel (a less dense, light yellowish liquid) by draining from the bottom of the funnel. Excess mixture of methanolic NaOH solution was mixed with the FAME in a separating funnel and stirred again for 30 minutes, after which the mixture was kept for 24 hrs to gradually separate under gravity. The mixture separated into two phases. The top segment contained of methyl esters and a little quantity of impurities like glycerol, residual alcohol, and partial glyceride, while the bottom segment consisted of glycerol. The methyl ester produced was continually washed with distilled water to remove the trace amounts of impurities present such as glycerol, residual methanol and remaining catalyst. $Na_2SO_4$ was used to remove residual water from the esters, and then filtered using a whatman filter paper (N0-42). First, the catalyst residue in the biodiesel layer was neutralized by adding of sulphuric acid solution to the pH level of 6.8—7.0 as recommended by [9].

During the washing process, gentle agitation was employed to avoid emulsion formation. The end product, methyl ester was obtained as a clear amber-yellow liquid. The methyl ester was then transferred to a clean dry flask to which silica gel was already added. The mixture was thoroughly stirred and then filtered to ensure complete removal of water, the methyl ester. The dry biodiesel obtained was placed into a pre-weighed brown bottle, weighed and kept for characterization and analyses.

**Oil and Biodiesel Characterization**

The physicochemical properties of the raw oil and the esters were computed in accordance to ASTM D6751-02. Percentage yield was determined using methods recommended by [10] cloud point, kinematic viscosity (viscometer), flash point (closed cup method), pour point by ASTM D2500, ASTM 1984, ASTM D93, ASTM D97 respectively. Density and specific gravity was determined using a density bottle. Moisture content, saponification value, percentage free fatty acid, moisture content as described by [11]. The acid value and elemental composition (%) (Ca, S, Na and P levels) as stated by the official procedures of analysis as designated by the Association of Official Analytical Chemists AOAC, 1990.

Iodine value by Wiji's method, total glycerol AOAC 1997, methyl esters profile using GC-MS machine.

## IV. Results and Discussion

Results of the physicochemical analysis of oil is given in Table 1, and for methyl ester in Table 2.

Table 1. Physicochemical Properties of Oil Obtained from Citrillus lantanus Seed Oil

| Parameters | Values |
|---|---|
| Yield (%) | 45.50 |
| Density (g/cm$^3$) @ 20$^0$C | 0.92 |
| Moisture content (%) | 0.54 |
| Viscosity at 40$^0$C (cSt) | 26.41 |
| Ash content (%) | 0.48 |
| Pour point | -7.80 |
| Free fatty acid (%) | 9.00 |
| Acid value (mgKOH/g) | 20.20 |
| Saponification value (mgKOH/g) | 201.61 |
| Iodine (g$I_{2/}$100g) | 101.52 |

**Designing and Analysis of Complex Process**

Design of statistical experiment is the process whereby an experiment is being planned and performed in such a way that maximum amount of data is obtained with least number of runs [13].

To obtain good results from an experimental design, six steps are involved: selection of process, variables, levels of varying of independent variables, explanation of factorial program, carrying out the experiment based on the experimental matrix, and obtaining values of response parameters. [14] which must be analysed to be optimized. An optimal response means generally a minimal or maximal value.

**Design of Experiment**

JMP SAS V14.0 was used in the design of experiment and development of regression model. To reduce the effects of uncontrolled factors, the experimental arrangement was randomised. Custom design with 4-factors and 1-response was applied with 2$^{nd}$ interactions, 2$^{nd}$ power and 6 centre

Table 2. Comparison of Fatty Acid Composition (wt%) of Oils from Different Seeds [12] and Values Obtained from this Work

| Literature | | | | | | | | This Work |
|---|---|---|---|---|---|---|---|---|
| Fatty Acids | Class | Palma | Jatropha | Rapeseed | Safflower | Soybean | Sunflower | Citrillus lantanus |
| Palmitic | C16:0 | 43.9 | 18.22 | 2.7 | 6.6 | 10.3 | 6 | 10.72 |
| Stearic | C18:0 | 4.9 | 5.14 | 2.8 | 3.3 | 4.7 | 5.9 | 7.7 |
| Oleic | C18:1 | 39 | 28.46 | 21.9 | 14.4 | 22.5 | 16 | 0 |
| Linoleic | C18:2 | 9.5 | 48.18 | 13.1 | 75.5 | 54.1 | 71.4 | 65.64 |
| Erucic | C22:1 | 0 | 0 | 50.9 | 0 | 0 | 0 | 0 |
| Total Saturated | | 51.2 | 23.36 | 5.5 | 10 | 15.1 | 12 | 20.91 |
| Total Unsaturated | | 48.8 | 76.64 | 94.5 | 90 | 84.9 | 88 | 56.22 |

 

points generating a total number of 28 runs. The independent variables (factors) were temperature (ºC), catalyst concentration (wt%), reaction time (min) and methanol-oil molar ratio (v/v), and the dependent variable (as response) was the yield computed using Eq (1). The level and ranges of the independent variables for the transesterification reaction is tabulated in Table (2). The center points consist of variables at zero level which are necessary for the reproducibility of the data and computation of experimental error. Each of the individual response gotten from the transesterification reaction was utilized in building a mathematical model that relates the independent reaction variables to the biodiesel yield through a 2nd-order polynomial equation as given in equation Eq. (1). The correlation value was used in determining the quality of the model developed, and analysis of variance (ANOVA) was used to determine the significance.

$$Y=b_o+\sum_{i=1}^{n} bixi+\sum_{i=1}^{n} bixi\ 2+\sum_{i=1}^{n-1} \quad \sum_{j=i+1}^{n} byxixi \qquad (1)$$

Where *bo, bi, bij bii* respectively represent the constant, linear, interactive coefficients, and quadratic coefficients, and *xi*, *xj* represent the coded values of the experimental variables. *Y* represents the predicted ester yield as the response.

Table 3. Factors and their Description

| | UNIT | LOWER LEVEL | UPPER LEVEL |
|---|---|---|---|
| Reaction temperature | (ºC) | 50 | 60 |
| Catalys concentration | (g) | 0.5 | 1.0 |
| Time of reaction | (min) | 30 | 120 |
| Methanol—Oil ratio | (ml) | 1:3 | 1:6 |

After designing and conducting the experiment, analyzing the experimental data follows. This stage consists of developing the Process Model and the Response Surface Methodology (RSM):

1. Process Model is a linear set of equation describing the results from experiment as a function of factor levels [13]. The model equation is most commonly in a linear or a quadratic form [15].

2. RSM is an assortment of statistical and mathematical analysis used in developing, refining and optimizing processes and products especially when multiple variables affect some output [16].

**Analysis of Experimental Results**

Table 4 displays the results of biodiesel yield obtained from the experimental design. From the table, the yield of biodiesel produced ranges from 61% to 85%.

Table 4. Full Factorial Experimental Design

| S/N | Temp | Cat. Conc. | Time | Meth:Oil | Yield |
|---|---|---|---|---|---|
| 1 | 50 | 0.5 | 30 | 1:3 | **0.61** |
| 2 | 50 | 0.5 | 75 | 1:4 | **0.72** |
| 3 | 50 | 0.5 | 120 | 1:6 | **0.79** |
| 4 | 50 | 1.0 | 30 | 1:5 | **0.75** |
| 5 | 50 | 1.0 | 120 | 1:3 | **0.62** |
| 6 | 50 | 1.5 | 30 | 1:4 | **0.72** |
| 7 | 50 | 1.5 | 30 | 1:6 | **0.72** |
| 8 | 50 | 1.5 | 75 | 1:3 | **0.81** |
| 9 | 50 | 1.5 | 120 | 1:5 | **0.84** |
| 10 | 55 | 0.5 | 30 | 1:4 | **0.85** |
| 11 | 55 | 0.5 | 120 | 1:5 | **0.83** |
| 12 | 55 | 1.0 | 75 | 1:3 | **0.70** |
| 13 | 55 | 1.0 | 75 | 1:4 | **0.84** |
| 14 | 55 | 1.0 | 75 | 1:5 | **0.80** |
| 15 | 55 | 1.0 | 75 | 1:6 | **0.76** |
| 16 | 55 | 1.0 | 75 | 1:6 | **0.76** |
| 17 | 55 | 1.0 | 75 | 1:6 | **0.80** |
| 18 | 55 | 1.5 | 30 | 1:5 | **0.66** |
| 19 | 55 | 1.5 | 120 | 1:4 | **0.82** |
| 20 | 60 | 0.5 | 30 | 1:5 | **0.60** |
| 21 | 60 | 0.5 | 30 | 1:6 | **0.61** |
| 22 | 60 | 0.5 | 75 | 1:3 | **0.67** |
| 23 | 60 | 0.5 | 120 | 1:4 | **0.79** |
| 24 | 60 | 1.0 | 30 | 1:3 | **0.81** |
| 25 | 60 | 1.0 | 120 | 1:5 | **0.82** |
| 26 | 60 | 1.5 | 75 | 1:4 | **0.83** |
| 27 | 60 | 1.5 | 120 | 1:3 | **0.71** |
| 28 | 60 | 1.5 | 120 | 1:6 | **0.80** |

**Development of Regression Model**

The mono-factorial experiments indicated that time of reaction, catalyst concentration, temperature, methanol/oil molar as the most significant variables in transesterification of Citrillus lantanus oil with sodium hydroxide catalyst. The most appropriate model to fit the response consistent with the successive model sum of square, was a quadratic-type model, which is attributable to the highest order polynomial and significance of added terms. In terms of coded factor for the production of biodiesel, the resultant equation is presented in Eq. (2):

$$\mathbf{Y = 0.0426 + 0.017X_1 + 0.0810X_2 - 0.00291X_3 - 0.0152X_4 + 0.00331X_2^2} \qquad (2)$$

where $X_1$ = temperature of reaction, $X_2$ = catalyst concentration, $X_3$ = reaction time and $X_4$ = Methanol-oil molar ratio.

ANOVA was performed further to define the impact and the fitness of the quadratic model, and resultant quadratic model is obtainable in Table 5. The Model F-value of 40.2583, Prob>F of < 0.00001 and R squared value of 0.920015 inferred that the model was significant at 90% sureness level as illustrated on Table 6. The Prob>F or p-value (probability of error value) is used to define the impact of each regression coefficient as well as effect of interaction of each cross-product. Smaller p-value depicts greater significance of the equivalent coefficient [17][18]. Here, the p-values show a value less than 0.05, meaning that the term was statistically significant. From the

 

ANOVA results in Table 4.8, T, C, S, M, and combination of M*M and C*S were significant terms in influencing the FAME yield. From Table 7, the lack of fit F-value of 2.5682 and p-value of 0.3172 (p-value > 0.05 is not significant) showed that relative to the pure error, the lack-of-fit is insignificant and the developed model is suitably fitted to the experimental data. The $R^2$ value of the model was obtained as 0.900023 signifying 90% of the total variation in the yield of biodiesel produced was credited to the process variables considered from the experiment. The closer the $R^2$ value gets to 1, the closer the predicted yield is to the actual experimental yield, and the more accurate the model will be.

Table 1. Analysis of Variance

| Source | DF | Sum of Squares | Mean Square | F Ratio |
|---|---|---|---|---|
| Model | 6 | 0.17528082 | 0.029213 | 40.2583 |
| Error | 21 | 0.01523867 | 0.000726 | **Prob > F** |
| C. Total | 27 | 0.19051950 | | <.0001* |

Table 2. Summary of Fit

| | |
|---|---|
| $R^2$ | 0.900023 |
| R Adj | 0.886162 |
| Root Mean Square Error | 0.025838 |
| Mean of Response | 0.771629 |
| Observations (or Sum Wgts) | 28 |

**Optimum Operating Conditions**

After successfully fitting the design to the problem and developing the prediction model equation, the optimum operating conditions were determined while maximizing desirability as 59.1°, 75min, 1.0 g/g, 1:3 for temperature, and time of reaction, catalyst loading, and methanol-oil molar ratio respectively to give a corresponding yield of 77.13% of biodiesel as shown in Figure 1. The individual effects of the process parameters can be seen with respect to their desirability. Increase in reaction temperature from 50 – 60 °C increases the biodiesel yield from 75 to 80 %. Increasing catalyt loading from 0.5 to 1.5 wt/wt has a similar effect as the reaction temperature. Increase in reaction time, however, has a more significant effect than temperature and catalyst loading, as increase from 45 to 117 mins increases the yield from 60 % to 90 %. Methanol-oil molar ratio, on the other hand, showed a curved effect as against the linear effects of the other parameters. An increase from 3 to 4 methanol-oil ratio increased the yield from 75 to 80 %, which remained steady then showed a decline beyond 5. This demonstrates that further increase in methanol beyond the optimum negatively affects the yield of biodiesel.

Table 3. Parameter Estimates

| Term | Estimate | Std Error | t Ratio | Prob>\|t\| |
|---|---|---|---|---|
| Intercept | 0.8174963 | 0.011767 | 69.48 | <.0001* |
| Temp.(50,60) | 0.0172292 | 0.006249 | 2.76 | 0.0118* |
| Cat. (0.5,1.5) | 0.0290797 | 0.006047 | 4.81 | <.0001* |
| Time (30,120) | 0.0855191 | 0.006247 | 13.69 | <.0001* |
| Meth:Oil (3,6) | 0.0103221 | 0.005799 | 1.78 | 0.0496 |
| Meth:Oil *Meth:Oil | -0.050905 | 0.013545 | -3.76 | 0.0012* |
| Catalyst *Time | -0.016516 | 0.006684 | -2.47 | 0.0221* |

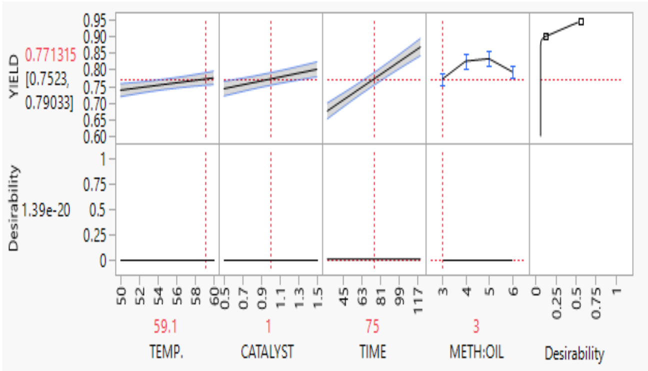


Figure 1. Prediction profile for optimum operating conditions

## V. Conclusion and Future Scope

The results of biodiesel production indicated that the maximum triglyceride conversion of 85% was attained at 55 °C temperature, 0.5 NaOH catalyst loading, 30 mins and 1:4 methanol oil ratio. The optimal operating conditions while maximizing desirability was found 59.1 °C, 1.0 g/g, 75 min, 1:3 respectively for temperature, catalyst concentration, time of reaction and methanol-oil molar ratio to give a corresponding yield of 77.13% of biodiesel. The optimum values of these parameters for attaining highest conversion of oil to esters is dependent on the physical and chemical properties of these oils. The biodiesel produced by alkali-transesterification reactions of Citrillus oil possesses qualities that meet ASTM standards. The profile of the plant's seed oil revealed the presence of major long chain fatty acids which are palmitic, stearic and linoleic as the major acid. Presence of saturation/unsaturation in the molecular structure of the corresponding free fatty acids in the oil decides both its physical and chemical behaviour. Therefore, Citrillus lantanus seed oil as a non-edible vegetable oil can be utilized to produce biodiesel. This will help reduce the competition between edible vegetable used for human consumption and energy production. Furthermore, the environment is cleaned by using renewable energy source in response to Sustainable Development Goals [20].

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