

Biodiesel Production from African Oil Bean Seed Oil: Process Optimization and Characterization Using Hybrid RSM–GA and ANFIS–GA Approaches

¹Ogwor E. Oghoghoreva, ²Elvis Emifoniye, ³Ogwor N. Dorathy, ⁴Ude C. Nonso

^{1,3}Department of mechanical engineering, Delta State Polytechnic Ogwashi-Uku. Nigeria.

²Department of Chemical Engineering, Delta State Polytechnic, Ogwashi-Uku. Nigeria.

⁴Federal University of Agriculture, Umudike. Nigeria.

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Abstract: Coal and natural gas are the primary fossil fuel sources that currently supply the majority of the world's energy needs. However, the World Energy Forum projects that these fossil-based resources could be depleted in less than a century. Consequently, the development of renewable alternatives to conventional diesel has become increasingly important. These alternatives must be technically feasible, economically and technologically competitive, environmentally acceptable, and readily available. Among the available options, biodiesel stands out as the most viable replacement for petroleum-based diesel fuel. In this study, transesterification was employed to produce biodiesel, as it is considered the most efficient and environmentally friendly method among existing production techniques. The feedstock used was African oil bean seed oil, from which Fatty Acid Methyl Esters (FAME), commonly known as biodiesel, were derived. To optimize biodiesel yield, five key process parameters were considered: Catalyst concentration: 0.775 wt% Methanol-to-oil molar ratio: 6.2:1, Agitation speed: 255 rpm, Reaction temperature: 60°C, Reaction time: 60 seconds. At these optimal conditions, the biodiesel yield reached 99.75%. The produced biodiesel was subsequently tested and characterized according to ASTM standards, and all measured properties fell within the specified standard limits.

Keywords: Biodiesel, Fatty Acid Methyl Esters (FAME), Transesterification, Optimization

I. Introduction

Energy is frequently cited as the key to a nation's progress. It is frequently used as a gauge to determine how fast a nation's economy is growing. In order to guarantee the sustainability of energy resources—both renewable and exhaustible—focused efforts are being made. Coal and natural gases are the primary sources of fossil fuels that supply the majority of the world's energy. The world's growing industrialization and motorization are driving up demand for petroleum-based fuels. Diesel fuel holds greater significance than all other fuels derived from petroleum. Diesel fuel is used in electric generators, city buses, locomotives, and other industrial sectors. It plays a crucial role in a nation's industrial economy. The petroleum-based diesel supply chain, however, has become unreliable due to a number of factors, including the high energy demand from various sectors, global warming, the unstable price and unavailability of petroleum-based oil, and environmental pollution from the widespread use of fossil fuels. The World Energy Forum has projected that natural gas and coal, among other fossil-based resources, will run out in less than a century [1]. As a result, developing renewable diesel resources to replace the conventional ones is becoming more and more important. Technically possible, economically and technologically competitive, environmentally acceptable, and easily accessible alternatives to diesel fuel are required [2]. Biodiesel is the name given to the substitute diesel fuel.

The American Society for Testing and Materials (ASTM 2022) defines bio-diesel as a fuel made of mono-alkyl esters of long-chain fatty acids that are sourced from either vegetable or animal fats [3], [4]. Biodiesel has the potential to meet the technical requirements of diesel fuel because of its strong molecular similarities to petroleum-based diesel [5]. With little or no adjustments, this clean-burning, renewable liquid fuel can be utilized in compression ignition engines in place of diesel derived from petroleum. The primary method of producing biodiesel, transesterification, is essentially reacting an alkyl-alcohol with a long-chain ester bond in the presence of a catalyst to produce glycerol and mono-alkyl esters, or biodiesel.

The terms *biodiesel* and *petroleum diesel* inherently describe their origins. Petroleum diesel is derived entirely from fossil fuels, whereas biodiesel is produced from renewable biological sources such as vegetable oil, peanut oil, coconut oil, neem seed oil, and even algal oil. In its raw form, biodiesel (B100) can be used directly as a fuel. However, unprocessed vegetable oils differ significantly from biodiesel in several key properties—they typically exhibit higher viscosity, higher volatility points, and lower oxidative stability. Despite these differences, many vegetable oils share several characteristics with petroleum diesel. As a result, biodiesel blends serve as a viable and immediate alternative energy source due to their favorable physical and chemical properties [6].

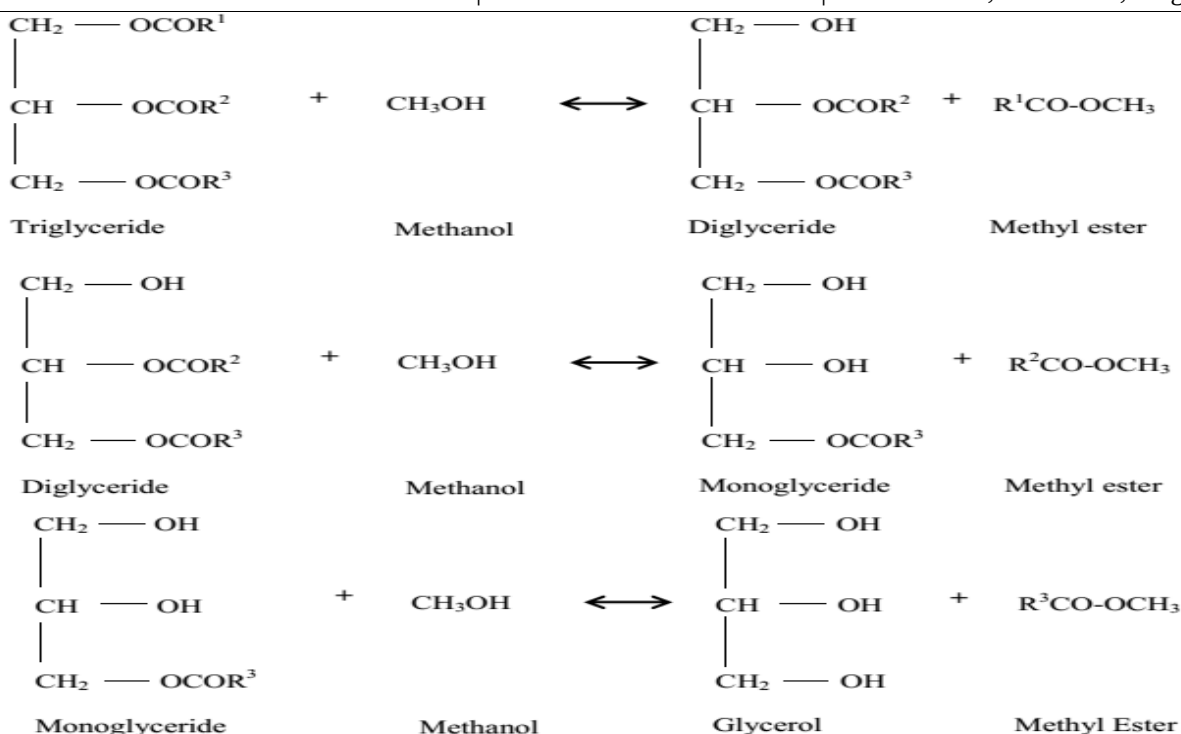


Fig 1: Detailed Step-by-step conversion of triglycerides to fatty acid alkyl ester (FAME) and glycerine.

Blends of biodiesel are utilized more frequently than pure B100 fuels. The main reason for this is because, biodiesel has a greater alcohol concentration than regular fuel, and running entirely on it occasionally necessitates engine changes. Fuel lines must now be steel due to these adjustments, since alcohol corrodes the more widely used rubber lines. Use of B99 (99% bio-diesel and 1% petroleum diesel) in place of B100 is widespread because only 1% of petroleum is poisonous enough to inhibit the growth of mold. The number following the letter "B" indicates the amount of biodiesel in the mix, and the letter "B" indicates the type of fuel—in this example, biodiesel. B5 is composed of 95% petroleum diesel and 5% biodiesel. B20 contains 80% petroleum diesel and 20% biodiesel in accordance with this guideline. By combining the two distinct diesel fuels, the fuel can benefit from biodiesel's lower emissions while also having a lower alcohol content, which enables the engine to function without any changes.

Rudolph Diesel invented the compression ignition engine using peanut oil one hundred years ago, vegetable oils have been used as an alternative fuel [7], [8], [9].

The cost of raw materials often accounts for the largest share of the total cost of producing biodiesel. According to reviews by [10], [11], and [12], 80–88% of the total cost of production or manufacturing is attributable to the usage of refined or high quality feedstock. Given the state of the economy today, rising refined oil prices are unavoidable and, as a result, account for an even larger portion of the cost of feedstock in the overall cost of production. The characteristics of the feedstocks also have a big influence on the final product's quality. According to [13], [14] and [15], a decrease in feedstock viscosity accelerates the rate of reaction and reduces its duration. Moreover, the quantity of oil produced by the crop determines the suitability of the feedstock. Higher oil yielding crops are better since they can lower production costs [16]. Vegetable oil (both edible and non-edible), animal oil and fats, and waste oil are the main categories of feedstocks used in the manufacturing of biodiesel [17]; [18]; [19]. The following is a discussion of the key raw materials and feedstocks.

Within the Leguminosea family, the African oil bean tree (*Pentaclethra macrophylla*) is indigenous to tropical Africa. It is well-known in Nigeria, where it goes by a number of names, including Apará in Yoruba for both the seed and the fermented product, with Ugba (Igbo) being the most well-known. Among consumers, it is a favourite condiment and meat substitute [20]. The eastern and southern regions of Nigeria are home to the tree. It is highly branched, reaching a height of roughly 21 metres and a girth of up to 6 metres, generating a canopy like a crown. It flowers in March and April, and then the mature, brown, woody pods burst open with an explosive mechanism, scattering the seeds as they curl up. The seeds are roughly 6 cm long and 3 cm wide, dorso-ventrally flattened, firm, and brown in colour [21]. About 47.90% of the seed is made up of oil [22].

Nevertheless, response surface methodology (RSM) has been used to investigate the optimisation of biodiesel production parameters from edible and non-edible oils. RSM is a reliable mathematical tool used in modelling, optimisation, and design of experiments (DoE) [23]. The primary advantage is in its capacity to decrease the quantity of experiments needed to assess the impact of various variables and their combined impacts on the outcome.

In terms of estimating capabilities, however, it is not as good as the adaptive neuro-fuzzy inference system (ANFIS) technique [24]; [25]. Fuzzy systems and neural networks are used to create ANFIS. It can potentially capture the synergistic advantages of both strategies because it combines the concepts of both into a single capsule [26]. These advantages include neural networks' processing capacity, fuzzy systems' ability to represent uncertainty, and easy learning procedures [27]. ANFIS has hardly ever been used in studies on the transesterification processes used to produce biodiesel, in contrast to many other fields of study. One such study [28] described the modelling of mechanically stirred and ultrasound-assisted palm oil transesterification to create biodiesel.

Increasing a process's efficiency requires first optimizing its operating process variables [23]. Since RSM can only locate local optima, it has an optimization algorithm, which is a local optimization approach, in contrast to most ANFIS modelling programs that lack built-in optimization tools [26]. But compared to other optimization techniques, it is less successful [25]. Generally, ANFIS models are optimized by combining them with global optimization techniques like genetic algorithms (GA). Reports on the utilization of ANFIS for transesterification process modelling are scarce.

Thus, the main goal of this work was to employ ANFIS-GA and RSM to optimize the process parameters for transesterification of oil bean and pawpaw seed oil.

II. Materials and Method

Materials

Local suppliers provided the African Oil Bean seeds. The analytical-grade chemicals that were obtained include methanol, n-hexane, sodium hydroxide, ethanol, chloroform, iodine, acetic acid, potassium iodine, starch indicator, sodium thiosulphate, HCl, chloroform, and sulphuric acid. A viscometer, magnetic hot plate, refractometer, conical flask, separating funnels, distillation column, were among the tools used in this project [3].

III. Methodology

Extraction of the oil from the seed

To get rid of contaminants and spoiled samples, the seeds were sun-dried and sorted. They were grated in an industrial blender after being sun-dried. Every grated sample was passed through a 500 µm laboratory sieve. 75g of the sieved material were weighed into cotton fabric and put into the 250ml Soxhlet extractor's thimble. A 500 ml circular flask with a flat bottom was filled with 250 ml of n-hexane. The condenser attached to the flat bottom round flask holding n-hexane was linked to the Soxhlet with the extraction thimble holding the sample in a semi-permeable membrane. A heating mantle was used to heat the Soxhlet extraction setup while water flow was permitted at the condenser's outer jacket. When all of the oil had been removed from the sample, the extraction process was stopped. While the oil and n-hexane combination in the flat bottom flask were separated by distillation, the defatted sample in the semi-permeable membrane was disposed of. While the oil was still in the flask, the n-hexane distilled over [3].

The percentage yield of the oil was calculated as thus:

$$\% \text{ yield} = \frac{\text{weight of the oil extracted}}{\text{weight of the sample used}} \times 100 \text{ [3]} \quad (1)$$

Pretreatment of the extracted oil

Because the extracted oil had a high free fatty acid (FFA) content—exceeding 1%—a pre-treatment process was necessary. To reduce the FFA level and prepare the oil for transesterification, acid-catalyzed esterification was performed using methanol and concentrated sulfuric acid as the catalyst. The process involved introducing the treated oil into a viscometer up to the “2d” mark and transferring it to the opposite side of the capillary tube. According to [30], the stopwatch was reset each time the oil was reintroduced into the original tube to maintain consistency during viscosity measurements. To eliminate moisture, the oil sample was initially heated on a heating mantle at 110°C for 10 minutes. Afterward, the heated sample was allowed to cool in a water bath to 60°C. A known weight of the oil was transferred into a 500 mL three-necked round-bottomed flask. Methanol (60 wt% of oil) and concentrated sulfuric acid (7 wt% of oil) were added to the flask. A reflux condenser was connected to the central neck of the flask, with water circulating through its outer jacket. A thermometer was inserted through one of the side arms to monitor the reaction temperature. The setup was mounted on a magnetic heating mantle and heated at 60°C for 120 minutes, with constant stirring at 450 rpm. After the reaction, the mixture was poured into a 150 mL separating funnel and allowed to settle into three distinct layers:

Top layer: Methanol

Middle layer: Pre-treated oil

Bottom layer: Water

The components were separated sequentially—water was drained first, followed by the oil, and finally the methanol. To purify the esterified oil, hot distilled water was added and the mixture was vigorously shaken, then allowed to settle. The formation of two clear layers—oil (top) and water (bottom)—was observed. The aqueous layer was removed through the separating funnel.

Finally, the pre-treated oil was oven-dried at 105°C until all residual water was removed. The dried oil was collected in a beaker and set aside for the transesterification process.

Production of Biodiesel by Transesterification Reaction

In the presence of NaOH, the oil sample will react with methanol to form glycerol and methyl esters of fatty acids, or biodiesel.

The reactor was a 500 ml three-necked round-bottom flask with a thermometer and sample exit on the side arms and a condenser on the central arm. The reactor's metal knob is rotated in an electromagnetic field by an electromagnetic hot plate, which also heats the reactor. A predetermined amount (50 g) of the oil sample was added to the flask, and the temperature inside the flask was raised to the reaction temperature (55°C). Next, the stirrer was turned on at a certain speed, marking this point as the zero time of the reaction, and methanol and the catalyst mixture (sodium methoxide) were introduced in the amount (0.5 wt%) defined (DoE) for the reaction. The reaction mixture was refluxed for the necessary reaction period of 45 minutes while being aggressively agitated at 300 rpm. The transesterified product was allowed to stand in a separating funnel for a day after the methanolysis process was completed, during which time it separated into an upper layer of biodiesel and a lower layer of glycerol. The top layer of biodiesel was tapped off after the bottom layer of glycerol.

Biodiesel purification

In a separating funnel, 1M sulphuric acid was applied dropwise to the biodiesel. Additionally, hot distilled water was added, and the mixture was shaken hard. When the mixture separated into two layers—the upper layer, which included the biodiesel, and the lower layer, which contained water and water-soluble contaminants—it was allowed to settle. After draining the water, three drops of phenolphthalein indicator were added. Washing was kept up until the waste water tested with phenolphthalein was clear and did not turn pink. After washing, the sample was dried by heating it on a hot plate in the lab to 105 degrees Celsius until all of the water molecules were gone.

Repeating the experiment at different temperatures, times, methanol-to-oil molar ratios, catalyst dosages, and agitation rates. Equation (2) is used for evaluating the biodiesel yield.

$$\text{Yield (\%)} = \frac{\text{weight of fatty acid methyl ester}}{\text{weight of oil}} \times 100 \quad (2)$$

Development of Models and Optimization

Table 1: Comparative Performance of RSM and ANFIS Models

Performance Characteristic	RSM	ANFIS
Modeling Approach	Based on second-order polynomial regression equations derived from statistically designed experiments (e.g., fractional factorial, central composite design).	Combines fuzzy logic and artificial neural networks to learn complex patterns in experimental data without assuming a fixed equation form.
Handling of Non-linearity	Handles moderate non-linearity through quadratic terms, but cannot fully capture highly non-linear, chaotic, or multi-modal relationships between process parameters and biodiesel yield.	Excellent at handling strong non-linearities, complex interactions, and non-additive effects between parameters.
Prediction Accuracy	High accuracy when the system truly follows a polynomial trend. However, accuracy drops when experimental data show strong non-linear deviations.	Typically achieves higher prediction accuracy for biodiesel yield because it adapts to complex, real-world reaction behavior.
Data Requirement	Works effectively with fewer data points due to structured experimental designs; good for initial exploration of factor effects.	Requires a larger dataset for training, testing, and validation to prevent overfitting and ensure generalization.
Interpretability	Highly interpretable: coefficients directly indicate how each variable and interaction affects biodiesel yield; statistical significance is clear.	Low interpretability: operates as a “black box” with fuzzy rules and membership functions that are not directly tied to physical or chemical meaning.
Optimization Capability	Identifies optimum reaction conditions within the tested range using regression equations; may miss global optima if surface is highly irregular.	Can find optima in highly non-linear systems and may detect patterns missed by polynomial models.

Experimental Design Integration	Integral part of Design of Experiments (DoE); provides both modeling and optimization in one framework.	Not inherently tied to DoE; relies on whatever dataset is provided, but can use RSM-designed data for better coverage of parameter space.
Computational Demand	Relatively low; involves regression fitting and simple optimization.	Relatively high; requires iterative training of membership functions and neural weights until convergence.
Sensitivity to Noise	Moderately sensitive; statistical regression smooths out noise, but extreme outliers can distort results.	Can be more sensitive to noise if not trained carefully, but robust fuzzy rules can mitigate this.
Overfitting Risk	Low if design and model assumptions are valid.	Higher risk if dataset is small or model complexity is too high; requires careful tuning.
Suitability for Mechanistic Insight	Strong: regression models reveal main effects, interaction effects, and quadratic effects, aiding understanding of reaction chemistry.	Weak: focuses on prediction accuracy rather than explaining mechanistic relationships.
Practical Use in Biodiesel Production	Excellent for initial screening of key process parameters (e.g., temperature, catalyst loading, methanol/oil ratio, reaction time, agitation speed).	Excellent for fine-tuning optimization and achieving higher yield predictions after key variables are identified.

Response surface methodology (RSM) of Design Expert version 6 was used to generate experimental design matrix for biodiesel production and model development. MATLAB 2013a was used for implementing the predictive models for adaptive neuro-fuzzy inference system (ANFIS) [3].

Experimental Design and Model Development by RSM [3]

Design Expert software (version 6) was used in this study to design the experiment and optimize the reaction conditions. The experimental design employed in this work was two-level-five factor fractional factorial design, including 32 experiments. Reaction temperature, catalyst concentration, methanol/oil molar ratio, reaction time and agitation speed were selected as independent factors for the optimization study. The response chosen was the methyl ester yields obtained from transesterification of pawpaw seed oil. Eight replications of centre points were used in order to predict a good estimation of errors and experiments were performed in a randomized order. The study of actual and coded levels of each factor and design matrix is shown in Tables 2. The coded values were designated by -1 (minimum), 0 (centre), $+1$ (maximum), $-\alpha$ and $+\alpha$. Alpha is defined as a distance from the centre point which can be either inside or outside the range, with the maximum value of $2^{n/4}$, where n is the number of factors. Hereby the value of alpha is set at 0.5 . It is noteworthy to point out that the software uses the concept of the coded values for the investigation of the significant terms, thus equation in coded values is used to study the effect of the variables on the response. The empirical equation is represented as shown in Equation (3)

$$Y = \beta_0 + \sum_{i=1}^5 \beta_i X_i + \sum_{i=1}^5 \beta_{ii} X_i^2 + \sum_{i=1}^5 \sum_{j=i+1}^5 \beta_{ij} X_i X_j \quad (3)$$

Selection of levels for each factor was based on the experiments performed to study the effects of process variables on the application of homogeneous base catalyst for transesterification reaction of the feed stocks.

Table 2: Studied range of each factor in actual and coded form [3].

Factor	Units	Low level	High level	$-\alpha$	$+\alpha$	0 level
Catalyst conc. (A)	Wt%	0.50(-1)	1.00(+1)	0.25(-2)	1.25(+2)	0.75
Methanol, (B)	Mol/mol	4(-1)	8(+1)	2(-2)	10(+2)	6
Temperature, (C)	°C	55(-1)	65(+1)	50(-2)	70(+2)	60
Reaction time (D)	Minutes	45(-1)	75(+1)	30(-2)	90(+2)	60
Agitation speed (E)	Rpm	200(-1)	300(+1)	150(-2)	350(+2)	250

Responses of Design Matrix for Optimization Study

Table 3: Responses of experimental design matrix for the transesterification of AOBSO [3]

Run order	Methanol/Oil molar ratio X ₁		Catalyst conc. (wt %) X ₂		Temperature (°C) X ₃		Time (Mints) X ₄		Agitation Speed (Rpm) X ₅		Biodiesel Yield Y (%)
	Coded	Real	Coded	Real	Coded	Real	Code d	Real	Code d	Real	
1	-1	4	-1	0.5	-1	55	-1	45	+1	300	59.00
2	+1	8	-1	0.5	-1	55	-1	45	-1	200	69.30
3	-1	4	+1	1	-1	55	-1	45	-1	200	67.00
4	+1	8	+1	1	-1	55	-1	45	+1	300	74.70
5	-1	4	-1	0.5	+1	65	-1	45	-1	200	55.90
6	+1	8	-1	0.5	+1	65	-1	45	+1	300	72.00
7	-1	4	+1	1	+1	65	-1	45	+1	300	67.00
8	+1	8	+1	1	+1	65	-1	45	-1	200	78.70
9	-1	4	-1	0.5	-1	55	+1	75	-1	200	60.00
10	+1	8	-1	0.5	-1	55	+1	75	+1	300	79.60
11	-1	4	+1	1	-1	55	+1	75	+1	300	72.20
12	+1	8	+1	1	-1	55	+1	75	-1	200	77.00
13	-1	4	-1	0.5	+1	65	+1	75	+1	300	67.20
14	+1	8	-1	0.5	+1	65	+1	75	-1	200	73.60
15	-1	4	+1	1	+1	65	+1	75	-1	200	65.20
16	+1	8	+1	1	+1	65	+1	75	+1	300	99.00
17	-2	2	0	0.75	0	60	0	60	0	250	26.00
18	+2	10	0	0.75	0	60	0	60	0	250	53.00
19	0	6	-2	0.25	0	60	0	60	0	250	74.70
20	0	6	+2	1.25	0	60	0	60	0	250	90.90
21	0	6	0	0.75	-2	50	0	60	0	250	85.70
22	0	6	0	0.75	+2	70	0	60	0	250	90.90
23	0	6	0	0.75	0	60	-2	30	0	250	79.60
24	0	6	0	0.75	0	60	+2	90	0	250	92.10
25	0	6	0	0.75	0	60	0	60	-2	150	80.70
26	0	6	0	0.75	0	60	0	60	+2	350	92.00
27	0	6	0	0.75	0	60	0	60	0	250	99.00
28	0	6	0	0.75	0	60	0	60	0	250	99.00
29	0	6	0	0.75	0	60	0	60	0	250	98.50
30	0	6	0	0.75	0	60	0	60	0	250	98.60
31	0	6	0	0.75	0	60	0	60	0	250	97.90

32	0	6	0	0.75	0	60	0	60	0	250	98.50
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Development of ANFIS Model

ANFIS combines the fuzzy logic and neural network approaches to create a hybrid intelligent model with advantages of both methods [31]. Figure 2 depicts the structure of an ANFIS model. ANFIS consists of five layers [31]. The first layer acquires the input parameters and introduces them to the ANFIS model. This layer is considered as the input of the fuzzy system. The output of the first layer becomes the input of the second layer carrying prior values of membership functions, MFs that are allocated based on the input values. The nodes on the second layer decide the fuzzy rules and send them to the third layer with a related degree of activity. The third layer normalizes the degree of activity for all rules. The fourth layer adopts the nodes and function and provides the first model by derived parameters [32] sending them to the output layer. The ANFIS model was developed by the ANFIS toolbox on MATLAB.

The input parameters were reaction temperature, catalyst concentration, methanol/oil molar ratio, reaction time and agitation speed and the only output parameter was methyl ester yield. The network was trained with three membership functions in order to select the type of membership function. The MF types include triangular-shaped, generalized bell-shaped, trapezoidal-shaped, and Gaussian membership functions. The output membership function type selected was the linear type because of its ability to further reduce errors. The training of ANFIS was done with a hybrid optimum method and an error tolerance value of zero.

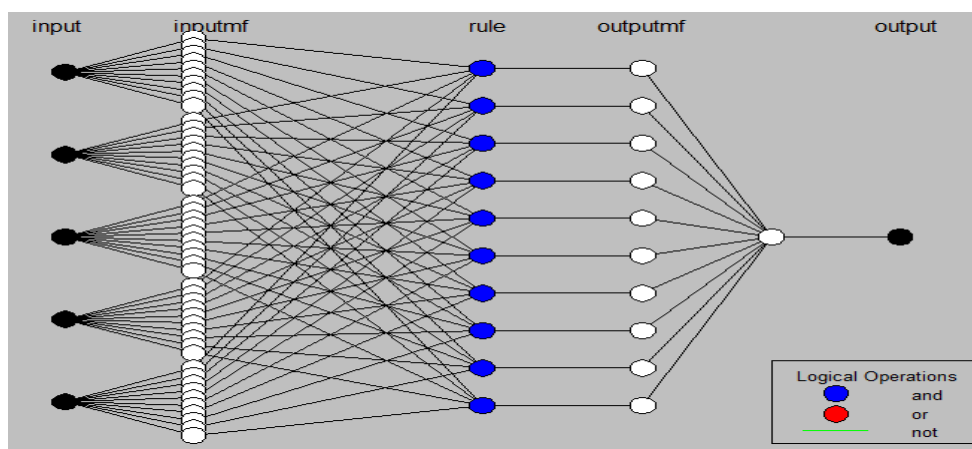


Figure 2: Architectures for model by ANFIS

Evaluation of developed models

The comparative performance of the RSM and ANFIS models was measured using statistical parameters, namely the root mean square error (RMSE), correlation coefficient(r), and the determination coefficient (R^2) as follows:

$$RMSE = \sqrt{\frac{1}{n} \sum_{i=1}^n (y_{p,i} - y_{a,i})^2} \quad (4)$$

$$R = \frac{\sum_{i=1}^n (y_{p,i} - y_{p,ave}) \cdot (y_{a,i} - y_{a,ave})}{\sqrt{[\sum_{i=1}^n (y_{p,i} - y_{p,ave})^2] [\sum_{i=1}^n (y_{a,i} - y_{a,ave})^2]}} \quad (5)$$

$$R^2 = 1 - \frac{\sum_{i=1}^n (y_{p,i} - y_{a,i})^2}{\sum_{i=1}^n (y_{p,i} - y_{a,ave})^2} \quad (6)$$

Where n = number of experiment data, $y_{a,i}$ is the experimental yield, $y_{p,i}$ is the predicted yield, $y_{a,ave}$ is the average experimental yield and $y_{p,ave}$ is the average predicted yield.

Optimization of transesterification process

In order to maximize the biodiesel yield, RSM and GA optimization tools were used to determine the best combination of the five process variables investigated.

For RSM optimization, the response (yield) was set at maximum while the process variables were set in the ranges studied. For GA optimization, the developed ANFIS and RSM models were individually coupled with the algorithm, and were then employed as the fitness functions [26]. The GA was used to search for the optimum values which gave the maximum yield. The main properties

of the GA used are presented in Table 4. The optimum values estimated by each method were validated by performing experiments in duplicate and the average values obtained were contrasted with the estimated values.

RSM optimization was carried out using Design Expert version 6.0 (Stat-Ease Inc., Minneapolis, MN, USA), and the GA tool kit in MATLAB R2013a (Mathworks Inc., Natick, MA, USA) was used for GA optimization.

Table 4: GA optimization parameters [3]

Genetic algorithm	Property	ANFIS	RSM
	Population	10	20
	Crossover	1/constraint dependent	1/intermediate
	Mutation rate	0.01/constraint dependent	0.01/uniform
	Generation	15-50	15-50
	Selection	Stochastic uniform	Stochastic uniform
	Creation function	Constraint dependent	Uniform

IV. Results and Discussion

OIL YIELD OF AFRICAN OIL BEAN

The oil yield from African oil bean seed using n-hexane is presented in Table 5. The percentage of the African Oil Bean Seed Oil (AOBSO) was 47.97 %, using n-hexane. The oil yield from African oil bean seed oil was significantly high. The yield was found to be comparable to some edible oil yields such as almond (47%); but they were also comparatively greater than the yields recorded for other non-edible seed oils *magnifera indica* (30.7%) [30]

J. Besides, the cost of producing biodiesel will be minimized, since the major feedstock is cheaply available, hence making biodiesel economically pleasant.

Table 5: Oil yields from pawpaw seed using n-hexane [3]

Type of Seed	Oil yield (%)
African Oil Bean Seed (AOBS)	47.97%

Characterization of Pawpaw Seed Oil

Physicochemical Properties of the Oils

Table 6 shows the physiochemical properties of the raw African oil bean seed oil. The oil has moderate acid number and free fatty acid values of 2.86 mgKOH/g and 1.43 %. These values suggest the pre-treatment step on the raw-oil before the transesterification step. The pre-treatment was able to reduce the FFA value below 1 %. The density and high viscosity of the oil will make its atomization difficult in internal combustion engine, hence it cannot be used directly as bio-fuel. The moderately low pour point of the raw and pre-treated oil shows that the oil will hardly solidify at room temperature hence can be stored for a long time. The high peroxide value of the raw oil was due to the presence of free radicals. The peroxide value of the raw African oil bean seed oil reduced after pre-treatment. The oxidation stability of the pre-treated oil was higher than the raw one and is good for production of biodiesel. This could be due to reduction in peroxide value and as a result of method used in extracting the oil. Solvent refining results in the production of base oil, which retain some sulphur compounds that are natural antioxidants. This base oil retain a natural ability to prevent oxidation, while hydro treated base oils must be further fortified with antioxidants in order to maintain thermal and oxidation stability.

Table 6: Physicochemical properties of African oil bean seed oil (AOBSO) [3]

S/N	Physicochemical properties	Raw AOBSO	Pre-treated AOBSO
1	Specific gravity	0.938	0.918
2	Acid value (mgKOH/g)	2.86	0.54
3	Free fatty acid (FFA) (%)	1.43	0.27
4	Spanofication value (mgKOH/g)	187.05	90.23

5	Iodine value (gI ₂ /100g)	36.49	36.49
6	Kinematic viscosity at 40°C (mm ² /s)	35.65	28.31
7	Peroxide value (meq/kg)	1.2	0.06
8	Flash point (°C)	329	305
9	Cloud point (°C)	14.50	20.60
10	Pour point (°C)	13	10
11	Moisture content (%)	10	7
12	Refractive index	1.47	1.46
13	Oxidation stability 11°C (Hour)	3	6
14	Molecular weight	901.06	901.06
15	Calorific value (MJ/g)	34.68	34.72

Modelling of the Transesterification Process and Optimization

The transesterification reaction was modelled using response surface methodology (RSM) and adaptive neuro fuzzy inference system (ANFIS).

Modelling using RSM

The responses of the experimental design matrix of transesterification of pawpaw seed oil (AOBSO) with methanol using NaOH are presented in Table 2. The data were fitted to the central composite design (CCD) second order mathematical equation. The equations which relates responses (AOBSO yield) to the process parameters in terms of the experimental values and coded forms, are described in Equations (7).

$$Y_{AOBSOB} = 98.58 + 6.85A + 4.03B + 1.26C + 3.13D + 2.78E + 0.35AB + 1.60AC + 1.18AD + 0.59AE + 1.14BC + 0.11BD + 0.37BE + 0.79CD + 1.2CE + 2.52DE - 14.76A^2 - 3.94B^2 - 2.56C^2 - 3.18D^2 - 3.05E^2 \quad (7)$$

Where Y_{AOBSOB} represents African oil bean seed oil biodiesel yield, A = catalyst concentration, B = methanol/oil molar ratio, C = Temperature, D = time and E = agitation speed.

The statistical significance of the mathematical regression models was tested using analysis of variance (ANOVA) and presented in Tables 7 for African oil bean seed oil biodiesel yield. From the table, it was observed that the model was significant at the 95% confidence level; the p value and F value of the models were <0.0001 and 4886.89. The level of significance of each parameters using ANOVA at 95% confidence level showed that all the linear terms, interactive terms except BD for the AOBSOB and all quadratic terms were significant. The insignificant terms were removed from the models and they reduced to Equation 7.

$$Y_{AOBSOB} = 98.58 + 6.85A + 4.03B + 1.26C + 3.13D + 2.78E + 0.35AB + 1.60AC + 1.18AD + 0.59AE + 1.14BC + 0.37BE + 0.79CD + 1.2CE + 2.52DE - 14.76A^2 - 3.94B^2 - 2.56C^2 - 3.18D^2 - 3.05E^2 \quad (8)$$

The performance of the mathematical models were evaluated using statistical indicators and the results obtained were: $R^2 = 0.9999$; adjusted $R^2 = 0.9997$. The predicted R^2 and adjusted R^2 were in reasonable agreement. These indicate good correlation between the experimental and predicted values as well as good fit of the model. [27] reported that a good model should have at least $R^2 = 0.8$. Adequate precision, which measures the signal to noise ratio, were 297.763. A ratio greater than 4 is required; thus, the model can be used to navigate the design space. The coefficient of variance (CV) was 0.39% for the models. The lower the CV, the smaller the residuals relative to the predicted value. The low CVs obtained indicated good models.

Table 7: Significance of regression coefficients of the yield of biodiesel produced from AOBSO using the design-expert version 6

Source	Sum of Squares	Df	Mean Square	F-value	p-value	
Model	8994.97	20	449.75	4886.89	< 0.0001	Significant
A-Catalyst conc.	1126.14	1	1126.14	12236.44	< 0.0001	
B-Methanol/Oil molar ratio	388.82	1	388.82	4224.80	< 0.0001	

C-Temperature	38.00	1	38.00	412.92	< 0.0001	
D-Reaction time	235.63	1	235.63	2560.28	< 0.0001	
E-Agitation speed	184.82	1	184.82	2008.17	< 0.0001	
AB	1.96	1	1.96	21.30	< 0.0001	
AC	40.96	1	40.96	445.06	< 0.0001	
AD	22.09	1	22.09	240.03	< 0.0001	
AE	5.52	1	5.52	60.01	< 0.0001	
BC	20.70	1	20.70	224.95	< 0.0001	
BD	0.20	1	0.20	2.20	0.1661	Not significant
BE	2.25	1	2.25	24.45	< 0.0001	
CD	9.92	1	9.92	107.82	< 0.0001	
CE	24.01	1	24.01	260.89	< 0.0001	
DE	102.01	1	102.01	1108.42	< 0.0001	
A ²	6394.62	1	6394.62	69482.85	< 0.0001	
B ²	455.31	1	455.31	4947.28	< 0.0001	
C ²	192.96	1	192.96	3217.61	< 0.0001	
D ²	296.2	1	296.2	21.30	< 0.0001	
E ²	273.28	1	273.28	445.06	< 0.0001	
Residual	1.01	11	0.6846			
Lack of Fit	0.18	6	0.031	0.19	0.9683	Not significant

Std. Dev. = 0.30, Mean = 77.95, C. V. = 0.39%, PRESS = 6.09, $R^2 = 0.9999$, Adj. $R^2 = 0.9997$, Pred. $R^2 = 0.9993$, Adeq Precision = 297.763.

The actual and predicted plots are shown in Figure 3. It could be seen from the figure that the data points on the plot were linearly distributed in all the catalyst used, indicating a good relationship between the experimental and predicted values of the response, and that the underlying assumptions of the above analysis were appropriate. The result also suggests that the selected quadratic model was proper and adequate in predicting the response variables for the experimental data.

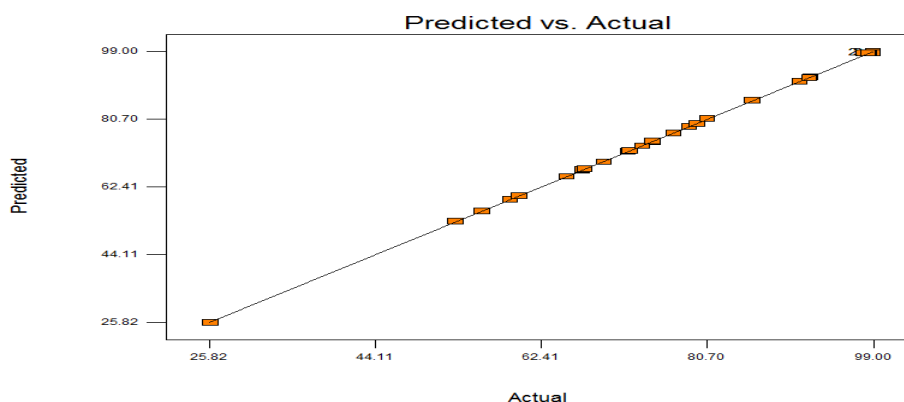


Figure 3: Plot of predicted values against the experimental values of biodiesel from AOBSOB

Figures 4 to 12 are the surface plots of the predicted biodiesel yield which can be generated by Equations 7 for the oil. The synergistic effect of catalyst conc. and methanol/oil molar ratio on yield of biodiesel is shown in Figure 4 for transesterification of AOBSO. The figures show that the amount of methyl ester yield increases with methanol/oil molar ratio and catalyst concentration. However, at higher catalyst concentrations and methanol/oil molar ratio, a reduction in the yield can be observed due to the fact that the quadratic terms of the two factors are more significant with a negative effect. The interaction effect of catalyst concentration

and temperature on yield of biodiesel is significant in all the catalyst used and shown in Figures 5. The figure indicates that the yield of biodiesel increases with reaction temperature and catalyst concentration. However, at higher catalyst concentration and reaction temperature, a decrease in the yield can be observed due to evaporation of methanol at higher temperature.

The interaction effect of catalyst concentration and time on yield of biodiesel is shown in Figures 6. The figure indicates that increase in reaction time and catalyst concentration increases the yield of biodiesel. However, at higher reaction time and catalyst concentration, reduction in the yield can be observed due to the saponification reaction.

The interaction effect of catalyst concentration and agitation speed on yield of biodiesel is significant and shown in Figure 7. The figure shows that the biodiesel yield increased with catalyst concentration and agitation speed which may be as a result of a proper mixing.

The interaction effect of methanol/oil molar ratio and reaction temperature on yield of biodiesel is shown in Figures 8. The figure indicates that the amount of biodiesel yields increased with methanol/oil molar ratio and reaction temperature. At higher methanol/oil molar ratio and reaction temperature, a drop in biodiesel yield was observed due to the evaporation of methanol.

The interaction effect of reaction methanol/oil molar ratio and agitation speed on yield of biodiesel is shown in Figures 9. The figure shows that the biodiesel yield increased with methanol/oil molar ratio and agitation speed as a result of a positive significant effect of methanol/oil molar ratio and agitation speed interaction term on response. However, at higher methanol/oil molar ratio and agitation speed, a reduction in the yield can be observed due to reduction in the effective collisions between the molecules of the reactants as a result of higher speed.

The interaction effect of temperature and time on yield of biodiesel from AOBSOB is shown in figure 10. At lower temperature, below 60°C the yield increased with increase in time. However, at a temperature above 60°C, there was reduction in yield. This may be as a result of evaporation of methanol which inhibits the reaction.

The combined effect of reaction temperature and agitation speed on yield of FAME is shown in Figures 10. The figure indicates that the yield of biodiesel increased with reaction temperature and agitation speed. However, at higher reaction temperature and agitation speed, there was a decrease in biodiesel yield because of minimum effective collisions caused by high agitation and evaporation of methanol.

The interaction effect of time and agitation speed on yield of biodiesel is shown in Figure 12. The figure indicates that the yield of biodiesel increased with reaction time and agitation speed. However, at higher reaction time and agitation speed, there was a reduction in biodiesel yield which may be attributed to reversible reaction of transesterification resulting in loss of esters.

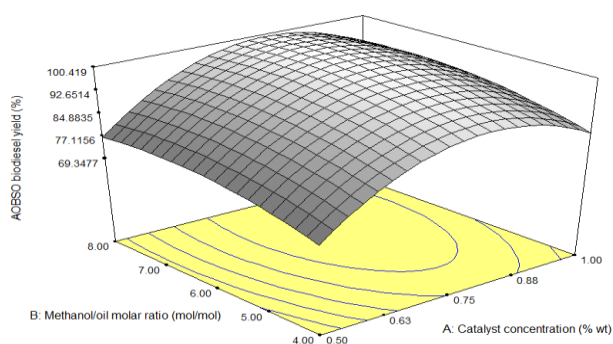


Figure 4: Interaction effect of catalyst conc. and methanol/oil molar ratio on AOBSO biodiesel yield

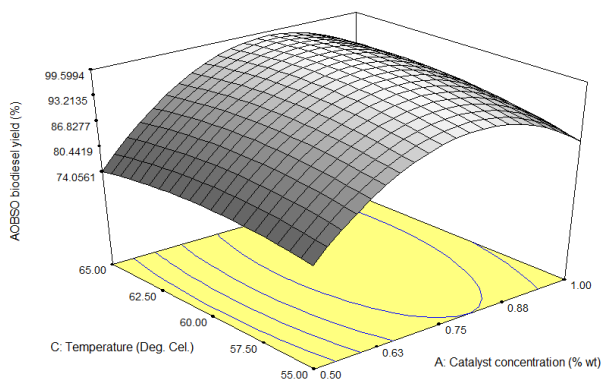


Figure 5: Interaction effect of catalyst conc. and temperature on AOBSO biodiesel yield

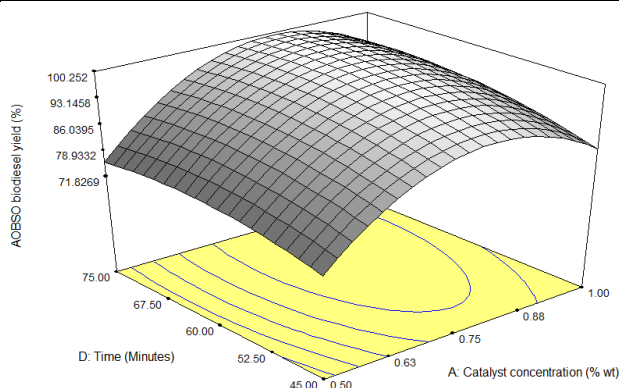


Figure 6: Interaction effect of catalyst conc. and time on AOBSo biodiesel yield

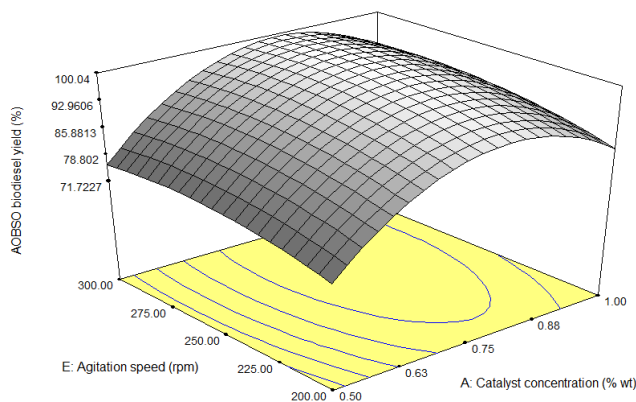


Figure 7: Interaction effect of catalyst conc. and agitation speed on AOBSo biodiesel yield

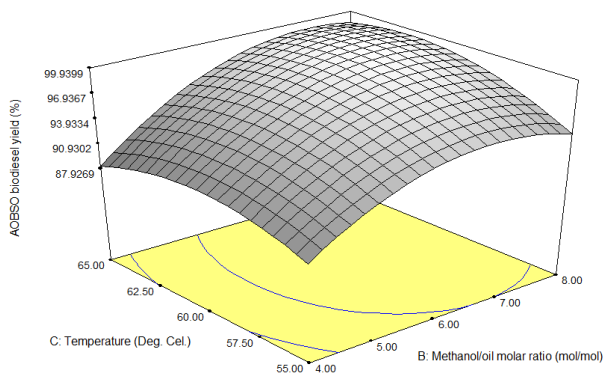


Figure 8: Interaction effect of methanol/oil molar ratio and temperature on AOBSo biodiesel yield

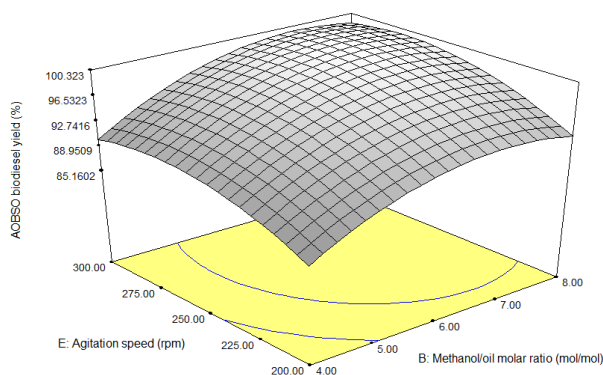


Figure 9: Interaction effect of methanol/oil molar ratio and agitation speed on AOBSo biodiesel yield

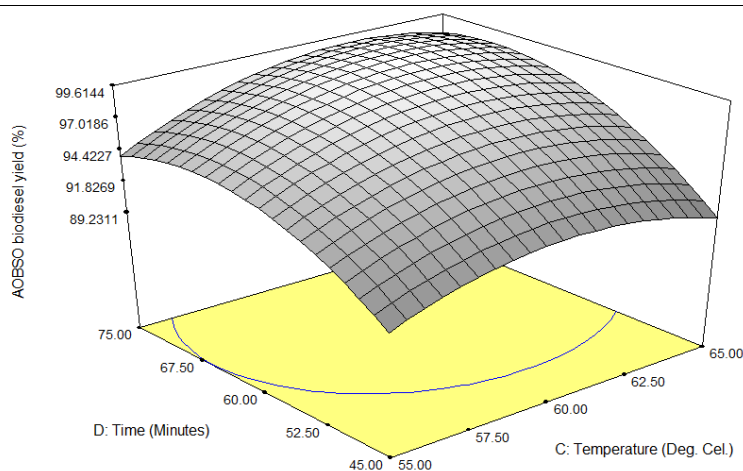


Figure 10: Interaction effect of temperature and time on AOBSO biodiesel yield

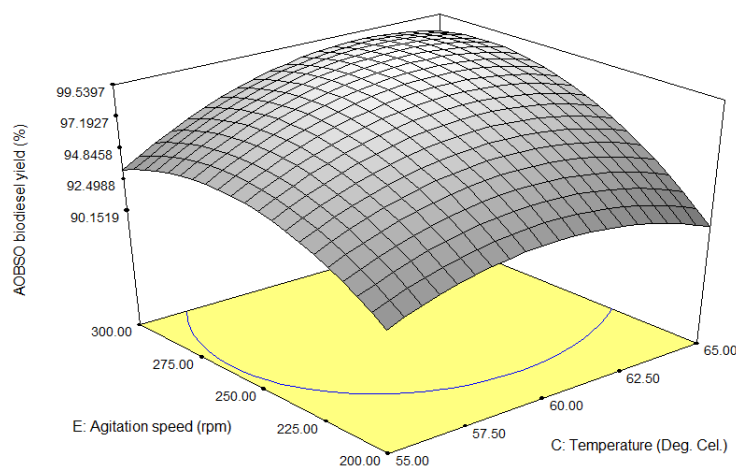


Figure 11: Interaction effect of temperature and agitation speed on AOBSO biodiesel yield

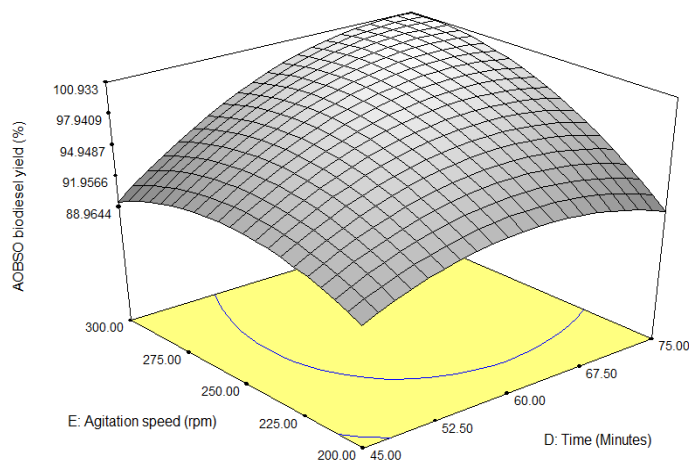


Figure 12: Interaction effect of time and agitation speed on AOBSO biodiesel yield

Modelling using ANFIS

The responses of the experimental design matrix of transesterification of African oil bean seed oil with methanol, using NaOH catalyst presented in Tables 2 were subjected to ANFIS model.

To illustrate the correctness of the model, Figure 13 shows the link between experimental and predicted values for the ANFIS model of transesterification of AOBSO biodiesel. Figures 13 illustrates the model's R^2 and R values, which were 0.9969; and 0.9984 respectively, with root mean square error (RMSE) values of 0.16089. A strong association between the experimental and predicted

values was supported by the high values of these statistical indices and the decreased RMSE. 99.2% of the variability between the experimental and projected values can be explained by the model.

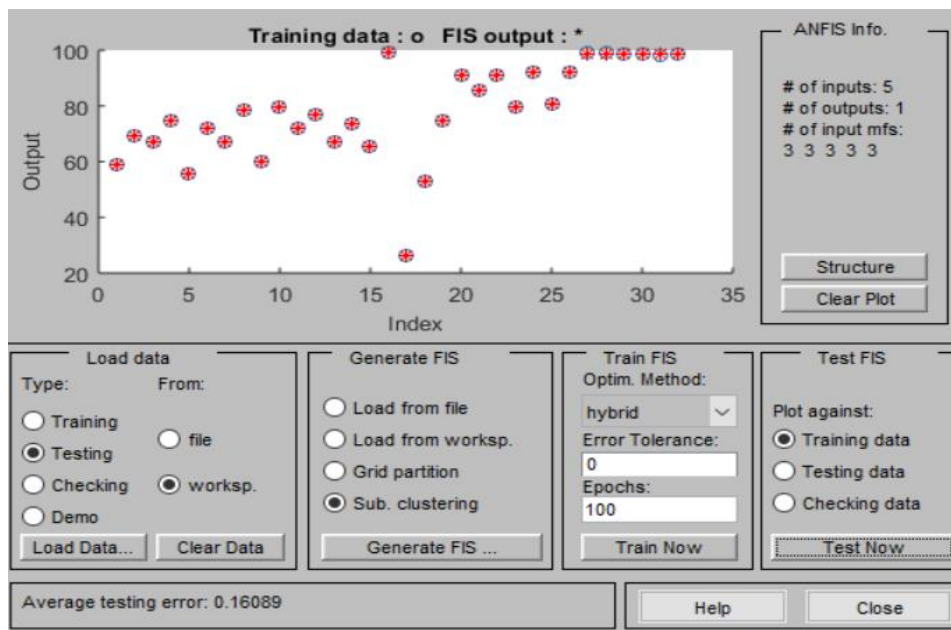


Figure 13: Experimental and estimated values against number of runs for AOBSOB ANFIS model.

Figures 14 to 22 are the three dimensional surface plots of the predicted biodiesel yield which can be generated by the ANFIS model for the African Oil Bean Seed oil (AOBSO).

The interactive effect of catalyst concentration and methanol/oil molar ratio on yield of biodiesel is shown in Figures 14 for transesterification of AOBSO. African oil bean seed oil biodiesel (AOBSOB) was significantly affected by the interaction between the two parameters. The figure shows that the amount of methyl ester yield increases with methanol/oil molar ratio and catalyst concentration. However, at higher catalyst concentrations and methanol/oil molar ratio, a reduction in the yield can be observed and this could be as a result of soap formation.

The interaction effect of catalyst concentration and temperature on yield of biodiesel is significant and shown in Figure 15. The figure indicates that the yield of biodiesel increases with reaction temperature and catalyst concentration. However, at higher catalyst concentration and reaction temperature, a decrease in the yield can be observed due to evaporation of methanol at higher temperature.

The interaction effect of catalyst concentration and time on yield of biodiesel is shown in Figures 16. The figure indicates that increase in reaction time and catalyst concentration increases the yield of biodiesel. However, at higher reaction time and catalyst concentration, reduction in the yield can be observed due to the saponification reaction.

The interaction effect of catalyst concentration and agitation speed on yield of biodiesel is significant and shown in Figure 17. The figure shows that the biodiesel yield increased with catalyst concentration and agitation speed which may be as a result of a proper mixing.

The interaction effect of methanol/oil molar ratio and reaction temperature on yield of biodiesel is shown in Figure 18. The figure indicates that the amount of biodiesel yield increased with methanol/oil molar ratio and reaction temperature. At higher methanol/oil molar ratio and reaction temperature, a drop in biodiesel yield was observed due to the evaporation of methanol. The interaction effect of methanol/oil molar ratio and agitation speed on yield of biodiesel is shown in Figure 19. The figure shows that the biodiesel yield increased with methanol/oil molar ratio and agitation speed. However, at higher methanol/oil molar ratio and agitation speed a reduction in the yield can be observed due to reduction in the effective collisions between the molecules of the reactants as a result of higher speed.

The interaction effect of reaction temperature and time on yield of biodiesel is shown in Figure 20. At lower temperature, below 60°C the yield increased with increase in time. However, at a temperature above 60°C, there was reduction in yield. This may be as a result of evaporation of methanol which inhibits the reaction.

The synergistic effect of reaction temperature and agitation speed on yield of FAME is shown in Figure 21. The figure indicates that the yield of biodiesel increased with reaction temperature and agitation speed. However, at higher reaction temperature and agitation speed, there was a decrease in biodiesel yield because of minimum effective collisions caused by high agitation and evaporation of methanol.

The interaction effect of time and agitation speed on yield of biodiesel is shown in Figure 22. The figure indicates that the yield of biodiesel increased with reaction time and agitation speed. However, at higher reaction time and agitation speed, there was a reduction in biodiesel yield which may be attributed to reversible reaction of transesterification resulting in loss of esters.

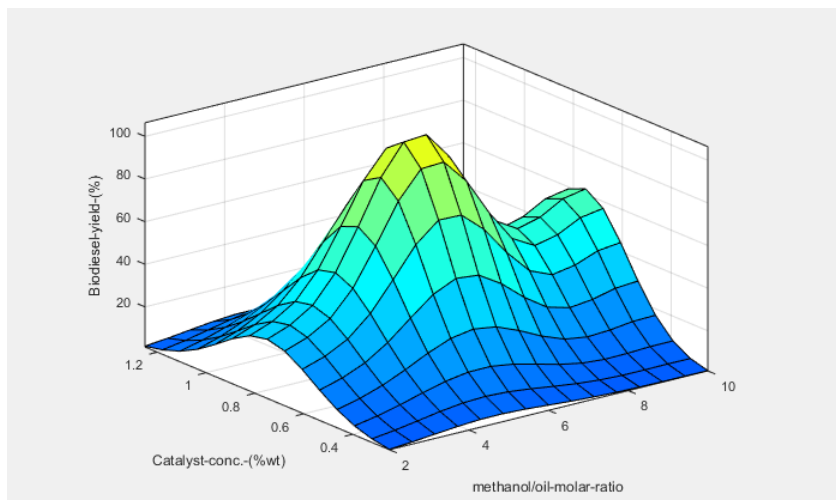


Figure 14: Interaction effect of catalyst concentration and methanol/oil molar ratio on AOBSo biodiesel yield

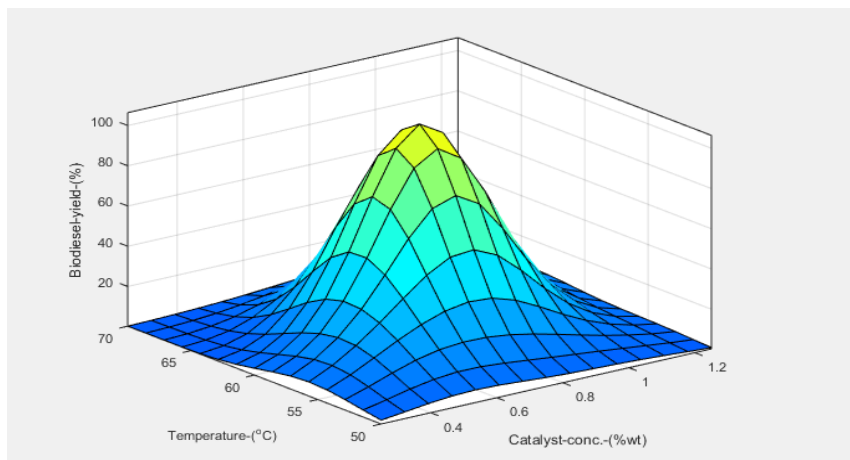


Figure 15: Interaction effect of catalyst concentration and temperature on AOBSo biodiesel yield

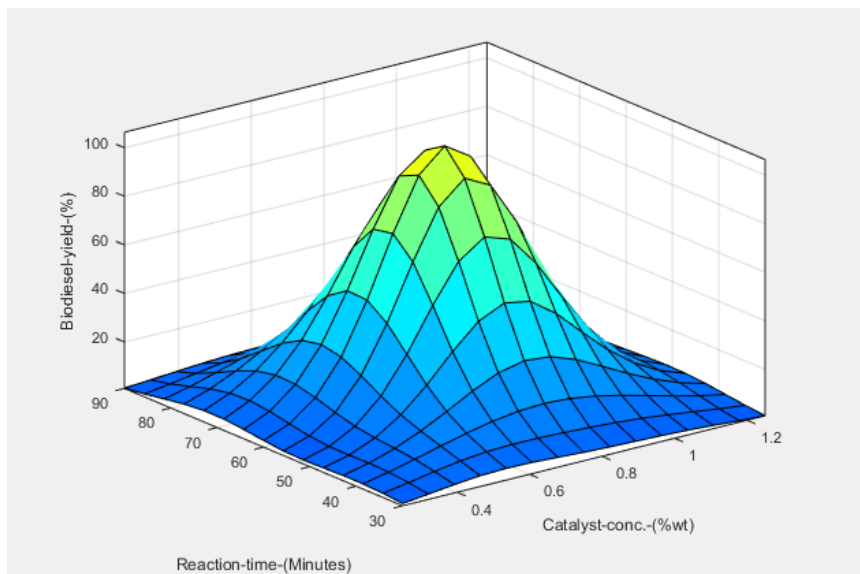


Figure 16: Interaction effect of catalyst concentration and time on AOBSo biodiesel yield.

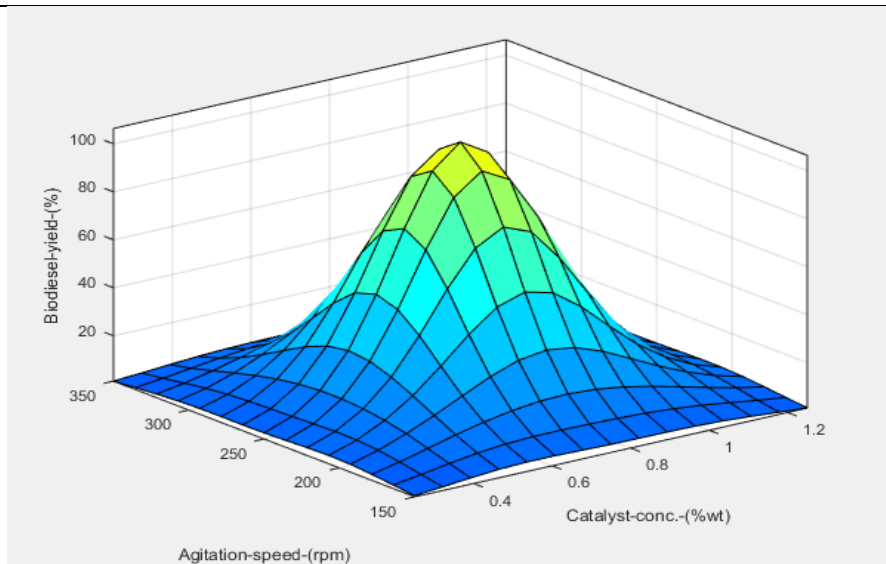


Figure 17: Interaction effect of catalyst concentration and agitation speed on AOBSO biodiesel yield.

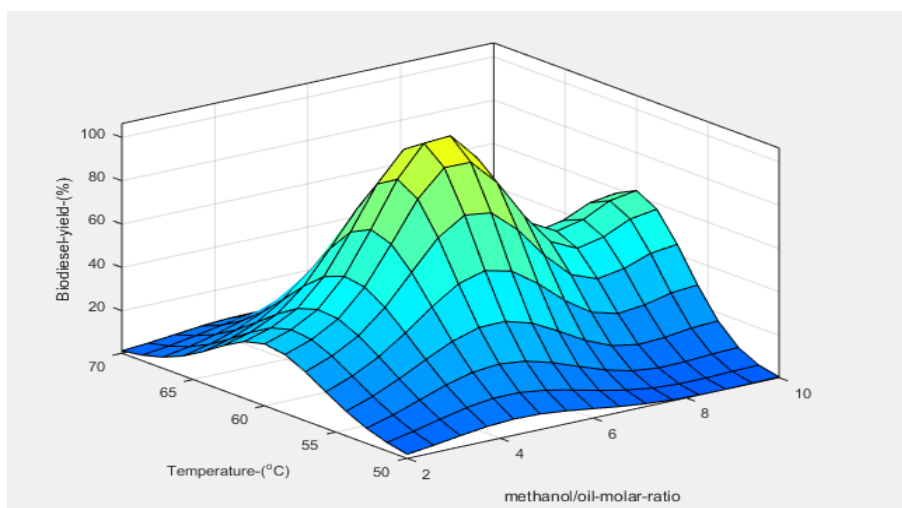


Figure 18: Interaction effect of methanol/oil molar ratio and temperature on AOBSO biodiesel yield.

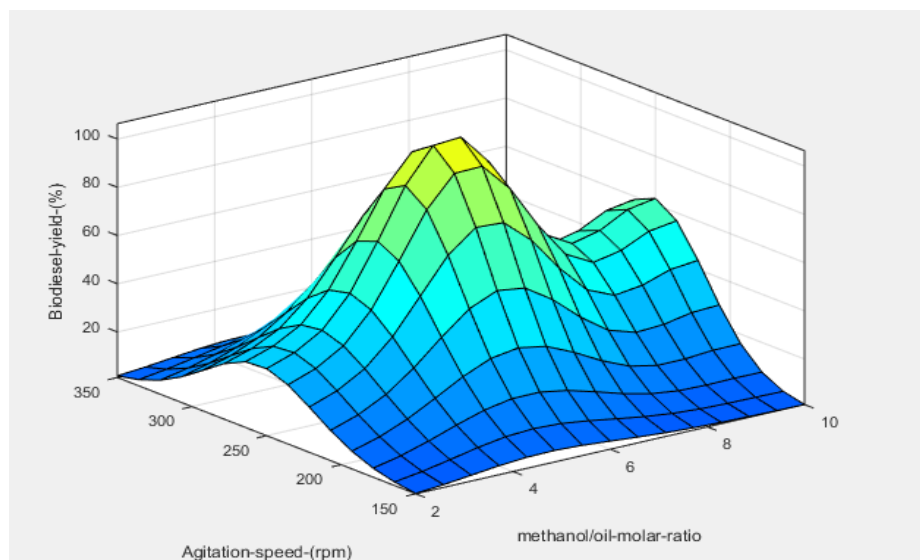


Figure 19: Interaction effect of methanol/oil molar ratio and agitation speed on AOBSO biodiesel yield.

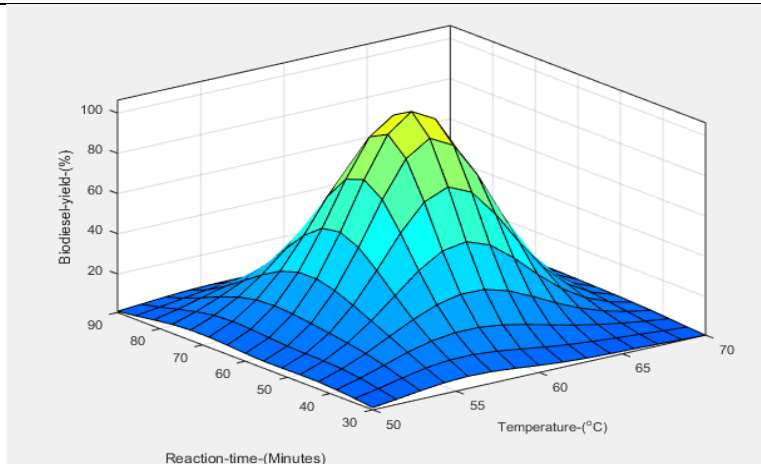


Figure 20 Interaction effect of temperature and reaction time on AOBSO biodiesel yield

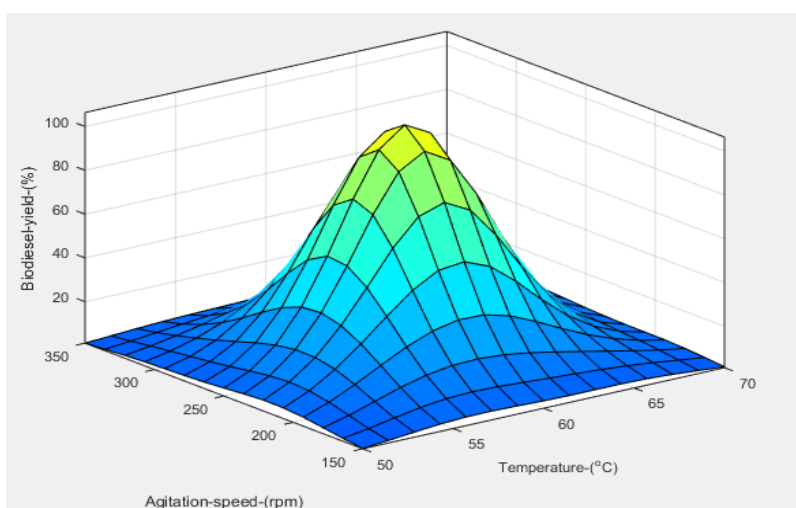


Figure 21: Interaction effect of temperature and agitation speed on AOBSO biodiesel yield.

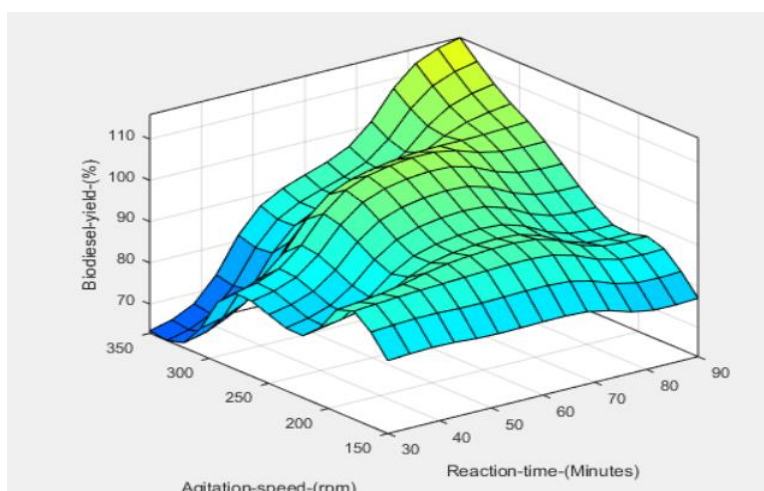


Figure 22: Interaction effect of reaction time and agitation speed on AOBSO biodiesel yield.

Environmental Impact

The African oil bean tree (*Pentaclethra macrophylla*) is a native of tropical Africa and belongs to the family *Leguminosea*. It is popular in Nigeria where it is known by several names such as *Apara* in Yoruba for the seed as well as the fermented product and the most prominent being *Ugba* (Igbo), as it is a popular condiment and meat analogue among consuming populations (Enujiugha and Akanbi, 2005). The tree thrives in the eastern and southern parts of Nigeria. It grows to a height of about 21 m, up to 6 m in girth and it is well branched, forming crown-like canopy. It

flowers between March and April after which the pods (brown and woody when matured) open by explosive mechanism, dispersing the seeds and curls up. The seeds are dorso-ventrally flattened, hard, brown in colour and about 6 cm long and 3 cm wide (Achinewhu *et al.*, 1998). The seed contains about 47.90% of oil (Ikhuoria *et al.*, 2008). The biodiesel from AOBSO is biodegradable, the risk of water contamination is low. Because it can be grown locally, over dependence on fossil fuel import is reduced. AOBSOB is eco-friendly because of its low carbon content, compared with fossil fuel carbon content. African Oil Bean Seed biodiesel emits little or no sulphur and its oxides into the atmosphere.

V. Conclusion

The biodiesel was produced from African oil bean seed oil by transesterification, and was characterised using ASTM (American Standard for Testing and Materials). African oil bean seed was the feedstock, and was sourced from local farmers. Methanol was the alcohol, n-hexane was the solvent, sodium hydroxide catalyst was used. Others include ethanol chloroform, iodine, acetic acid, potassium iodine, starch indicator, sodium thiosulphate, HCl, chloroform, and sulphuric acid. A viscometer, magnetic hot plate, refractometer, conical flask, separating funnels, distillation column, were among the tools used in this project. The process parameters were reaction time, reaction temperature, agitation speed, catalyst concentration, and methanol/oil molar ratio.

RSM-GA, ANFIS-GA and RSM produced nearly the same yields for AOBS oil biodiesel, with RSM-GA producing the best yield. The catalyst concentration of 0.775%wt, methanol/oil molar ratio of 6.2:1, agitation speed of 255 rpm, temperature of 60°C and reaction time of 60 minutes were the optimal parameters for RSM-GA, and the biodiesel yield was 99.75%.

The characterization of the biodiesel produced under optimized conditions met the limits of ASTM 6751 standards.

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