

Research Article

Comparism of Response Surface Methodology (RSM) and Adaptive Neuro-Fuzzy Inference Systems (ANFIS) in Optimisation of Soybean Soapstock Biodiesel Production

Chinedu Gabriel Mbah ^{*} , Francisca Unoma Nwafulugo, Njideka Ophelia Ezetoha

Chemical Engineering Department, Federal Polytechnic, Oko, Nigeria

Abstract

Soybean soapstock (SS), a lipid rich by-product of soybean oil production is a promising feedstock for the production of biodiesel due to its availability and affordability. In the esterification and transesterification reactions involving soybean soapstock, sodium hydroxide, methanol and n-hexane were used as catalyst, solvent and co-solvent respectively. The physico-chemical properties of the biodiesel obtained were determined using the Association of Analytical Chemist (AOAC) and American Society of Testing Materials (ASTM) methods. The esterification and transesterification reactions were optimised using both response surface methodology (RSM) under design expert 7.0 platform and Particle swarm technique in ANFIS (ANFIS-PSO) using the MATLAB software. The optimized acid value from the esterification reaction using RSM and ANFIS-PSO were 4.956 and 1.488 while the yield obtained were 97.29% and 99.91% respectively with ANFIS-PSO proving to be the better optimization technique in both cases. Comparison plots made for both reactions shows the ANFIS-PSO curve mirroring the experimental and thus signifying a closer trend when compared to the RSM curve. The suitability of the ANFIS-PSO prediction was further highlighted by the error analysis carried out on both techniques. The Residual sum of squares (RSS), Mean absolute error (MAE), Root mean square error (RMSE), Correlation coefficient (R), Coefficient of determination (R^2), Adjusted R^2 , Absolute average deviation (AAD) and Mean absolute percent error (MAPE) values for the ANFIS-PSO predictions in both reactions were better than the RSM predictions. It can thus be concluded that soybean soapstock is a viable feedstock for biodiesel production and ANFIS-PSO is a more efficient optimization technique when compared with RSM in esterification and transesterification of soybean soapstock.

Keywords

Biodiesel, Soybean Soapstock, Response Surface Methodology (RSM), Adaptive Neuro-Fuzzy Inference System (ANFIS), Particle Swarm Optimization (PSO)

1. Introduction

Renewable energy has become a major research focus due to decreasing fossil fuel reserves and climate implications of their use in the transport and industrial sector [10]. Biodiesel,

a non-toxic biodegradable fuel has over the years, proven to be a very reliable source of renewable fuels. Biodiesels are generally monoalkyl esters of long chain fatty acids derived

*Corresponding author: gadoral@yahoo.com (Chinedu Gabriel Mbah)

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from renewable feedstock like vegetable oils and animal fat through esterification and trans esterification reactions [11]. Trans esterification is normally carried out using bases such as sodium hydroxide and potassium hydroxide as catalysts, while alcohols such as methanol and alcohol are used as solvents due to their availability, affordability and potency in transesterification reactions [4]. To optimize a dependent variable such as yield in transesterification reactions, process parameters such as temperature, time, catalyst concentration, methanol/oil ratio and speed can be varied to ascertain their effect on the process [5]. Response surface methodology (RSM) as an optimization technique can be applied in several fields such as chemical engineering process control and chemical analysis among many other applications [3] and can be used to determine the optimum conditions in esterification and transesterification reactions. RSM uses regression and correlation analysis to evaluate the effect of two or more independent factors on the dependent variables. Awolu & Layokun, [2] and Kalil et al, [7] described RSM as a good optimizer involving a collection of statistical techniques for designing experiments, building models, evaluating the effects of factors and searching for the optimum conditions. For input data that are ambiguous or subject to a relatively high uncertainty, a hybrid fuzzy system such as adaptive neuro-fuzzy interference system, (ANFIS) may be a better optimization option when compared to other techniques such as RSM [8]. ANFIS, an adaptive network is a network structure that connects several nodes to several links. The nodes represent processing units and the links show the connection between those processing units. The rules of learning are made in a way to reduce system error and properly correct the node parameters. To determine the parameters, the ANFIS uses the hybrid learning principle, which combines the method of gradient descent and the least squares method [9]. This paper highlights the soybean soapstock biodiesel production process and carried out a comparative study of the use of both RSM and ANFIS-PSO as optimization techniques for the process.

2. Materials and Methods

2.1. Reagents and Equipment

The reagents used were methanol (Sigma-Aldrich), sodium hydroxide (NaOH) flakes, phenolphthalein, sulphuric acid, magnesium trisilicate, sodium sulphate, n-hexane and diethyl ether. Among the equipment used were a centrifuge (used for separation of soapstock from water and impurities), electronic weighing balance (B. Bran Scientific, England), heat drying oven (DHG Series Ocean Med+ England), electronic temperature regulation heating mantle (98-I-B Series), HH-S thermostatic water bath (DKS Series; Ningbo Biocotek Scientific Instrument Co. Limited, gas chromatography coupled FID and ECD (for obtaining fatty acid profiles) and buck scientific infra-red spectrophotometer (for characterizing of

the samples). All thereagents were of the required analytical standard and obtained from Springboard research laboratories, Awka, Anambra State, Nigeria.

2.2. Sample Collection

Soybean soapstock which is a by-product of soybean oil processing plants was acquired from Sunchi farms, a feed processing plant in Eleme, Enugu State, Nigeria. The sample collected was separated into distinct layers by the use of a centrifuge. The top layer which is the acid oil (AO) is utilized for the biodiesel production.

2.3. Characterisation of Soybean Soapstock and Biodiesel (Gas Chromatography)

Gas chromatography/mass spectrometry were used to analyze the fatty acid composition according to AOAC procedures (AOAC 2000). Calibration of the gas chromatography was carried out using established biodiesel standards and n-hexane in ethyl acetate solution. Hydrogen at 41.27 ml/min flow rate was used as the carrier gas. Retention time and mass spectra were utilized in peak identification [6]. This was carried out on both feedstock and biodiesel eventually produced.

2.4. Production of Biodiesel

Esterification was carried out by mixing same quantity of soybean soapstock, and methanol with a sulphuric acid catalyst in the ratio of 1:10 to the solution mixed. The solution was then heated to 60°C for 80 mins. For transesterification, the oil realized from esterification was mixed with methanol and n-hexane in the ratio of 1:3:3 respectively. A 2% sodium hydroxide catalyst (NaOH) was used and the solution heated to 55°C for 50 mins. This process is followed by separation using a separating funnel where the bottom layer (biodiesel) is recovered from the top layer (glycerol).

2.5. Physico-Chemical Analysis

Some of the physico-chemical properties of the biodiesel produced and the standards used were, kinematic viscosity (ASTM-445), density (ASTM D-1298), pour point (ASTM D-97), flash point (ASTM D-93), cloud point (ASTM D-2500), acid value (D-664), calorific value (ASTM D-246) and sulphur content (D-4294). Other properties such as iodine value, specific gravity and refractive index were measured by AOAC methods.

2.6. Optimisation Using Response Surface Methodology (RSM)

In optimization using response surface methodology (RSM), a software (design expert) was used for experimental

design, model building and obtaining optimum conditions [Zahed]. Design expert utilizes multiple regression and correlation analysis as tools to evaluate the effects of two or more independent factors on dependent variables [2]. Box-behken and fractional factorial were used for esterification and transesterification reactions and is presented in Table 1 and Table 2 respectively. The consequences of adjusting pro-

cess variables were monitored from the 3D plots generated. Deviations of the values predicted with the actual were obtained using regression analysis and analysis of variance (ANOVA). The fitted polynomial equations obtained from the regression analysis were then used to generate a ramp of optimized values.

Table 1. Factors and their levels of CCD for esterification.

Variables/Unit	Symbols	Coded			levels	
		-2	-1	0	+1	+2
Catalyst concentration (wt%)	A	5	10	15	20	25
Methanol/FFA volume ratio	B	2:1	4:1	6:1	8:1	10:1
Temperature (°C)	C	55	60	65	70	75
Esterification time (min)	D	60	70	80	90	100

Table 2. CCD levels of independent variables for experimental design of Base transesterification.

Independent variables	Symbols	Coded			Levels	
		- α	-1	0	+1	+ α
Temperature (°C)	X ₁	45	50	55	60	65
Reaction time (min)	X ₂	45	50	55	60	65
Catalyst concentration (wt %)	X ₃	0.50	1.00	1.50	2.00	2.50
Methanol/oil ratio (mol/mol)	X ₄	3:1	4:1	5:1	6:1	7:1
Stiring speed (rpm)	X ₅	200	300	400	500	600

2.7. Optimisation Using ANFIS-PSO

Optimisation was carried out using Particle swarm technique in ANFIS on the MATLAB software platform. Fuzzy inference system is based on the concept of fuzzy set theory, fuzzy if-then rules and fuzzy reasoning. The fuzzy inference engine is responsible for the evaluation of fuzzy rules to produce an output for each rule [1]. Interactive effects of adjusting the process variables were monitored using 3D surface and contour plots from the experimental runs made. The MATLAB software trains the system to assume a trend, generating predicted values for the objective variable using the ANFIS command in the fuzzy control toolbox. Particle swarm optimization technique was used to predict the opti-

mum values.

3. Results and Discussion

3.1. Optimisation of Soybean Soapstock Acid Value Using RSM

Reaction time, temperature, catalyst concentration and methanol/oil ratio were all important factors in the esterification reaction of soybean soapstock. The interactive effects of adjusting the process variables within the design space were monitored using 3D surface plots presented in Figure 1a-d on the Design Expert 7.0.0 platform.

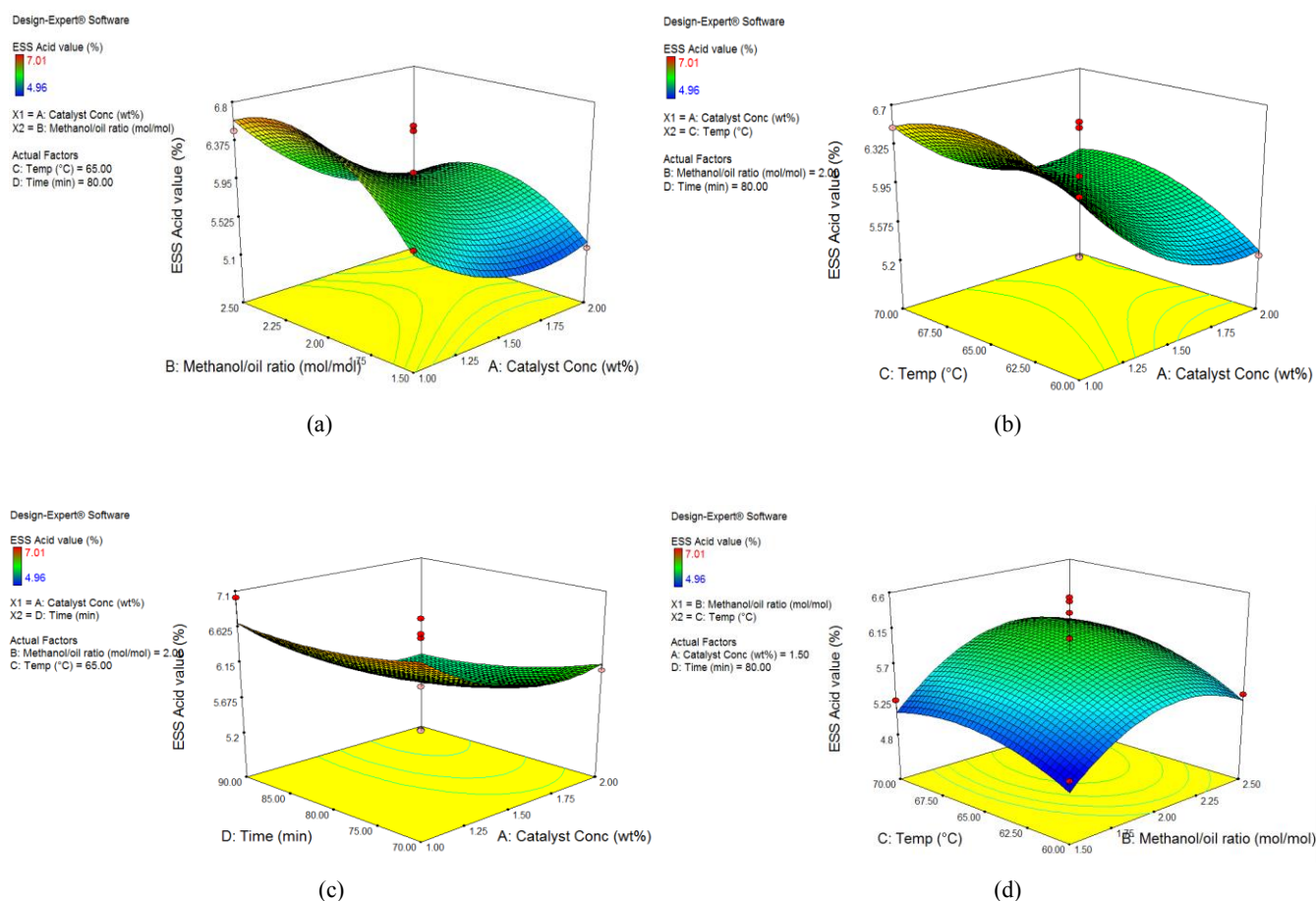


Figure 1. The 3D response surface plot of the effects of some variables on Acid value.

Experimental runs carried out by a combination of the four variables resulting in a total of 29 experimental runs as presented in Table 3 below. The table presented both the acid values and predictions made. It was observed that run 19 had the lowest actual acid value of 4.96 from the following reaction parameters: catalyst concentration (1.5), methanol/oil ratio (1.5), temperature (60) and time (80). This acid value was considerably lower than the predicted value at that run. This however also shows that though RSM made good predictions on soybeansoapstockesterifications, it did not properly mirror the actual acid values and thus leaves room

for improvement on the predictions. On the other hand, run 13 had the lowest predicted acid value of 4.83 from reaction parameters: catalyst concentration (1), methanol/oil ratio (2), temperature (65) and time (90). Though this acid value was lower than the lowest actual acid value (4.96), the considerable difference in its corresponding (run 13) actual acid value (7.01) signifies the unsuitability of RSM as a prediction technique in the esterification of soybeansoapstock. The high standard deviation (5.43) and low adjusted R^2 values as seen in Table 5 further proves the unreliability of RSM as a prediction technique for esterification of soybeansoapstock.

Table 3. Esterification runs and corresponding RSM predictions.

Run	Catalyst. Concentration. (wt%)	Methanol/oil ratio (mol/mol)	Temperature	Time (min)	Acid Value	Rsm Prediction
1	1.5	2	70	90	5.67	5.76
2	1.5	2	65	80	6.03	5.25
3	2	1.5	65	80	5.18	6.6
4	1.5	1.5	65	70	5.04	5.49
5	1.5	2	65	80	6.54	5.86

Run	Catalyst. Concentration. (wt%)	Methanol/oil ratio (mol/mol)	Temperature	Time (min)	Acid Value	Rsm Prediction
6	1.5	2	60	90	4.99	5.98
7	1.5	2	60	70	5.98	5.32
8	2	2	65	70	6.06	5.97
9	2	2	70	80	5.76	6.78
10	2	2	60	80	5.25	6.15
11	1.5	2.5	65	90	5.35	6.68
12	1.5	2	70	70	6.12	5.69
13	1	2	65	90	7.01	4.83
14	1.5	1.5	70	80	5.25	5.25
15	2	2.5	65	80	5.26	5.09
16	1.5	1.5	65	90	5.3	5.76
17	1.5	2.5	70	80	5.87	6.28
18	1	1.5	65	80	5.8	5.3
19	1.5	1.5	60	80	4.96	6.5
20	1.5	2	65	80	5.24	5.86
21	1	2	60	80	6.32	5.09
22	1.5	2	65	80	5.23	6.33
23	2	2	65	90	6.22	5.51
24	1.5	2.5	60	80	5.33	5.36
25	1	2	70	80	6.48	5.91
26	1	2.5	65	80	6.48	5.91
27	1.5	2.5	65	70	6.48	5.91
28	1	2	65	70	6.48	5.91
29	1.5	2	65	80	6.48	5.91

The low F-value of 2.73 as seen in Table 4 indicates there is no significant difference between both groups. The smaller the P-value, the more reliable the prediction will be. The "Lack of Fit F-value" of 0.22 implies the Lack of Fit is not significant relative to the pure error. The "Pred R-Squared"

of 0.8752 is in reasonable agreement with the "Adj R-Squared" of 0.9131. "Adeq Precision" measures the signal to noise ratio. A ratio greater than 4 is desirable. The ratio of 6.296 indicates an adequate signal and can thus be used to navigate the design space.

Table 4. ANOVA for response surface quadratic soyabeansoapstock esterification model.

Source	Sum of squares	Df	Mean square	F value	p-value Prob> F	
Model	7.05	14	0.5	2.73	0.0014	significant
A-Catalyst Conc (wt%)	1.97	1	1.97	10.67	0.0056	
B-Methanol/oil ratio (mol/mol)	0.88	1	0.88	4.78	0.0463	
C-Temp (°C)	0.45	1	0.45	2.45	0.0397	

Source	Sum of squares	Df	Mean square	F value	p-value Prob> F	
D-Time (min)	0.22	1	0.22	1.21	0.2894	
AB	0.092	1	0.092	0.5	0.0011	
AC	0.029	1	0.029	0.16	0.6968	
AD	0.033	1	0.033	0.18	0.6801	
BC	0.016	1	0.016	0.085	0.7755	
BD	0.49	1	0.49	2.64	0.0023	
CD	0.072	1	0.072	0.39	0.543	
A^2	0.62	1	0.62	3.36	0.008	
B^2	1.26	1	1.26	6.84	0.0204	
C^2	0.35	1	0.35	1.89	0.191	
D^2	0.077	1	0.077	0.42	0.5288	
Residual	2.59	14	0.18			
Lack of Fit	0.93	10	0.093	0.22	0.9747	not significant
Pure Error	1.66	4	0.41			
Cor Total	9.64	28				

Table 5. Summary of soya soap stock esterification regression values.

Std. Dev.	5.43	R-Squared	0.9316
Mean	5.8	Adj R-Squared	0.9131
C.V. %	7.41	Pred R-Squared	0.8752
PRESS	117.95	Adeq Precision	6.296

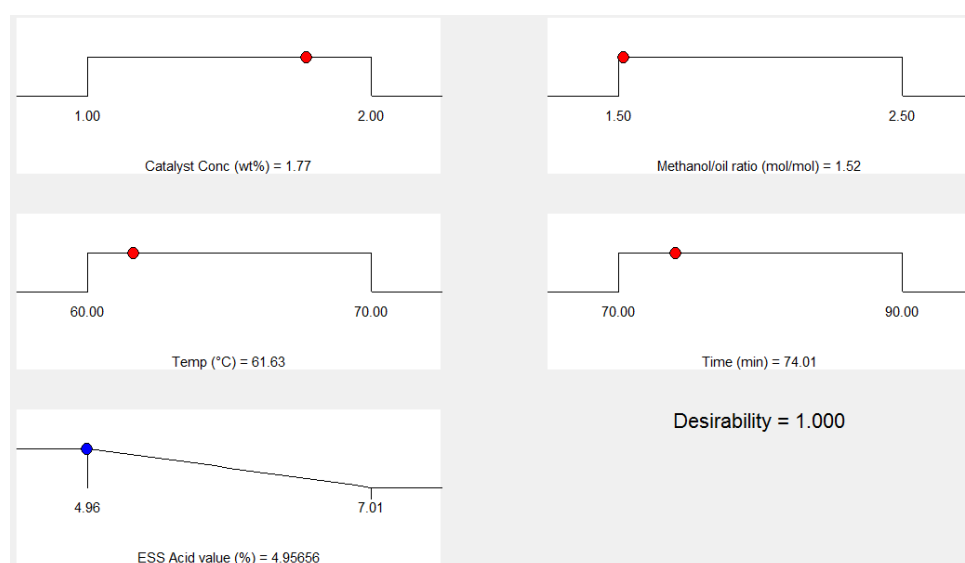


Figure 2. Ramps of the optimization of esterification of soya soap stock.

From the ramp of optimized values generated from equations obtained in terms of coded and actual factors in the optimization of the esterification of soya soap stock, the optimized acid value was 4.956, based on the outcome presented in Figure 2. it can also be concluded that the optimum parameters for the esterification process are: temperature (61.63°C), reaction time (74.01 mins), catalyst concentration (1.77 wt%) and methanol/oil ratio (1.52 mol/mol) with boundary condition of each factor also displayed in Figure 2.

3.2. Optimisation of Soybean Soapstock Biodiesel Yield Using RSM

Parametric effects of reaction variables such as reaction time, temperature, catalyst concentration, methanol/oil ratio and stirringspeed are all important factors in the transesterification of soybean soapstock. These interactive effects on soybean soapstock biodiesel yield were illustrated in Figure 3 below.

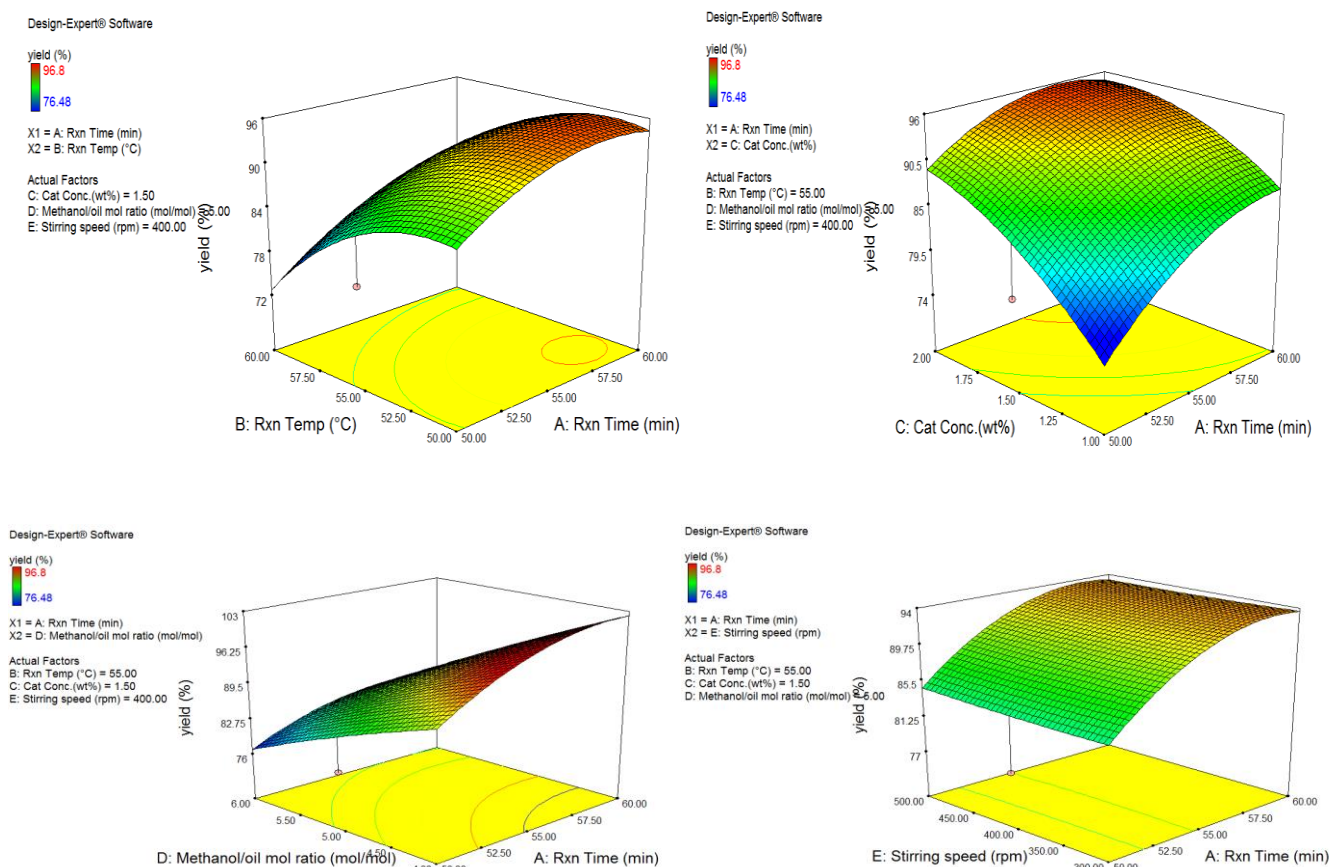


Figure 3. The 3D response surface plot of the effects of some variables on SSSME yield.

Table 6. Runs for transesterification of esterified soya soapstock using RSM.

Run	Time (min)	Temperature (°C)	Catalyst concentration (wt%)	Methanol/oil ratio (mol/mol)	Stirring speed (rpm)	Yield (%)	Rsm Prediction (%)
1	50	50	2	5	400	90.9	93.98
2	50	50	2	7	400	90.48	87.98
3	50	50	3	5	400	91.88	86.48
4	50	50	2	5	400	92.98	87.78
5	65	45	2.5	4	500	77.88	74.88
6	50	45	2.5	4	300	93.08	97.58

Run	Time (min)	Temperature (°C)	Catalyst concentration (wt%)	Methanol/oil ratio (mol/mol)	Stirring speed (rpm)	Yield (%)	Rsm Prediction (%)
7	65	55	2.5	6	500	77.48	75.08
8	50	45	2.5	6	500	93.18	95.38
9	50	45	1.5	6	300	79.38	77.48
10	50	55	2.5	6	300	79.38	89.48
11	50	50	2	5	400	93.78	97.48
12	50	50	2	5	400	91.28	95.18
13	65	55	2.5	4	300	93.98	93.08
14	45	50	2	5	400	78.88	79.38
15	65	55	1.5	4	500	95.08	97.38
16	50	50	2	5	400	95.48	93.48
17	50	50	2	5	600	95.38	97.48
18	50	50	2	5	400	95.18	97.48
19	50	50	1	5	400	79.48	85.08
20	50	55	1.5	4	300	96.8	95.88
21	65	45	2.5	6	300	77.98	79.9
22	50	55	1.5	6	500	78.48	81.28
23	65	45	1.5	6	500	77.58	73.58
24	50	60	2	5	400	78.48	75.78
25	50	50	2	5	200	93.98	94.98
26	50	55	2.5	4	500	93.58	93.98
27	50	45	1.5	4	300	79.48	83.18
28	50	50	2	3	400	93.08	95.88
29	65	55	1.5	6	300	76.48	79.48
30	50	55	1.5	5	400	77.78	78.88
31	50	45	2	5	400	94.98	92.98
32	55	45	2.5	4	500	78.88	86.8

Experimental runs were carried out by a combination of these five variables resulting in a total of 32 experimental runs as presented in Table 6 which shows the runs for the transesterification of soybeansoapstock and their respective actual and predicted yields. It was observed that the highest actual yield was at run 20 with reaction parameters: time (50 mins), temperature (55°C), catalyst concentration (1.5), methanol/oil ratio (4) and stirring speed (300 rpm) had a yield of 96.8%. This compared favorably with the corresponding predicted yield (95.88%). However, the highest

predicted yield of 97.58% was obtained at run 6 using reaction parameters: time (50 mins), temperature (45°C), catalyst concentration (2.5), methanol/oil ratio (4) and stirring speed (300 rpm) was considerably higher than the corresponding actual yield (93.08%). This discrepancy in actual and predicted yields and the high standard deviation values obtained (4.83) as seen from Table 7 however indicate the unsuitability of RSM as an efficient technique in optimization of transesterification of soybeansoapstock.

Table 7. ANOVA for the Soya soap stock transesterification response quadratic model.

Source	Sum of Squares	Df	Mean Square	F Value	p-value Prob> F	
Model	1627.63	20	81.38	3.49	0.0086	significant
A-Rxn Time (min)	144.16	1	144.16	6.19	0.0302	
B-Rxn Temp (°C)	94.17	1	94.17	4.04	0.0695	
C-Cat Conc.(wt%)	128.23	1	128.23	5.5	0.0088	
D-Methanol/oil mol ratio (mol/mol)	381.23	1	381.23	16.36	0.0019	
E-Stirring speed (rpm)	0.014	1	0.014	6.19E-04	0.9806	
AB	8.95	1	8.95	0.38	0.548	
AC	58.55	1	58.55	2.51	0.0012	
AD	29.9	1	29.9	1.28	0.0013	
AE	0.69	1	0.69	0.029	0.8668	
BC	16.62	1	16.62	0.71	0.4163	
BD	241.37	1	241.37	10.36	0.0082	
BE	10.16	1	10.16	0.44	0.0025	
CD	6.27	1	6.27	0.27	0.6143	
CE	0.48	1	0.48	0.021	0.8881	
DE	79.74	1	79.74	3.42	0.0913	
A ²	146.35	1	146.35	6.28	0.0022	
B ²	243.35	1	243.35	10.45	0.008	
C ²	142.4	1	142.4	6.11	0.0015	
D ²	9.18	1	9.18	0.39	0.5431	
E ²	0.53	1	0.53	0.023	0.8823	
Residual	256.28	11	23.3			
Lack of Fit	237.83	6	39.64	8.74	3.099	Not- significant
Pure Error	18.45	5	3.69			
Cor Total	1883.91	31				

Table 8. Summary of Soya soap stock transesterification regression values.

Std. Dev.	4.83	R-Squared	0.964
Mean	86.96	AdjR-Squared	0.9466
C.V. %	5.55	Pred R-Squared	0.9004
PRESS	420.9	Adeq Precision	5.474

The Model F-value of 3.49 as seen in Table 7 implies the model is significant. There is only a 1.86% chance that a

"Model F-Value" this large could occur due to noise. Values of "Prob> F" less than 0.0500 indicate model terms are significant. In this case A, C, D, AC, AD, BE, BD, A², B², C² are significant model terms. Values greater than 0.1000 indicate the model terms are not significant. If there are many insignificant model terms (not counting those required to support hierarchy), model reduction may improve your model. The "Lack of Fit F-value" of 8.74 implies the Lack of Fit is Not-significant relative to the pure error. There is only a 0.99% chance that a "Lack of Fit F-value" this large could occur due to noise. Non-significant lack of fit is good -- we want the model to fit. The "Pred R-Squared" of 0.9004 is in reasonable agreement with the "Adj R-Squared" of 0.9466. "Adeq Precision" measures the signal to noise ratio. A ratio

greater than 4 is desirable. The ratio of 5.474 thus indicates an adequate signal making this model suitable for navigating the design space.

From the optimization of the transesterification of the soya soap stock, with an optimized yield of 97.29%, it can thus be

concluded that the optimum parameters for the process are: temperature (50.68°C), reaction time (53.36 mins), catalyst concentration (1.77 wt%), methanol/oil ratio (4.36 mol/mol) and stirring speed (407.60 rpm).

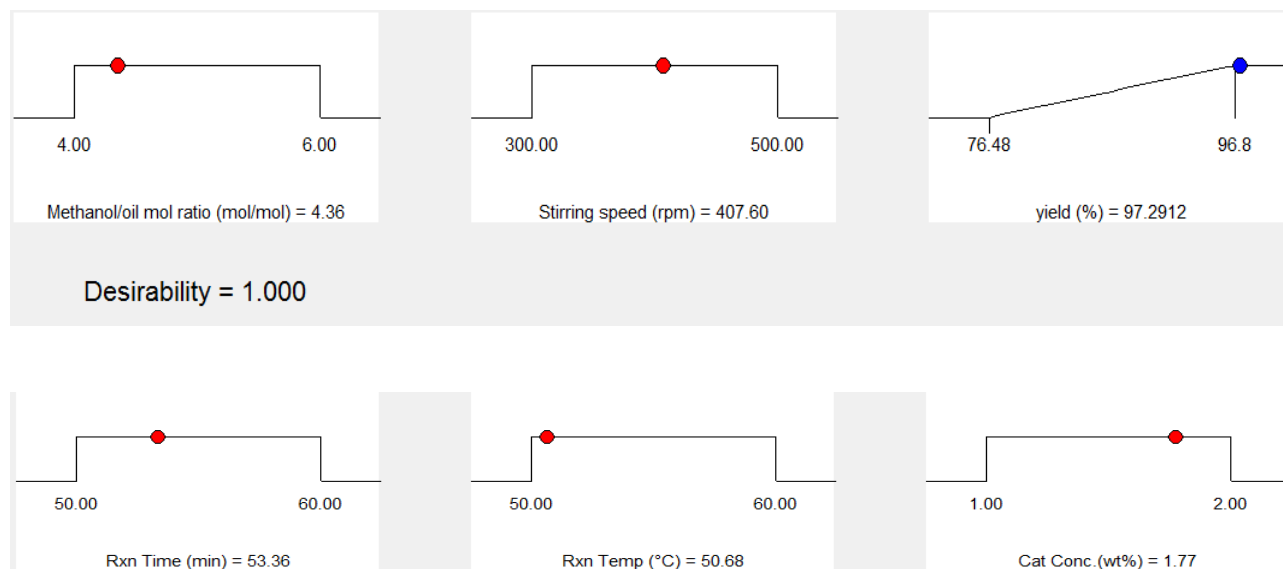


Figure 4. Ramps of the optimization of trans esterification of soya soap stock.

3.3. Optimisation of Soybean Soapstock Acid Value Using ANFIS

3D and contour plots were used to study the effect of the factor interaction of reaction time, temperature, catalyst concentration, and methanol/oil ratio in ANFIS. ANFIS predictions for the 29 runs were made and compared with values obtained from actual runs as shown in Table 9.

Table 9. Runs for esterification of soyabeansoapstock using ANFIS.

Runs	Catalyst concentration (wt%)	Methanol/oil ratio (mol/mol)	Temperature (°C)	Time (min)	Acid Value	ANFIS prediction
1	1.5	2	70	90	5.67	5.67
2	1.5	2	65	80	6.03	5.904
3	2	1.5	65	80	5.18	5.18
4	1.5	1.5	65	70	5.04	5.04
5	1.5	2	65	80	6.54	5.904
6	1.5	2	60	90	4.99	4.99
7	1.5	2	60	70	5.98	5.98
8	2	2	65	70	6.06	6.06
9	2	2	70	80	5.76	5.76
10	2	2	60	80	5.25	5.25
11	1.5	2.5	65	90	5.35	5.35
12	1.5	2	70	70	6.12	6.12

Runs	Catalyst concentration (wt%)	Methanol/oil ratio (mol/mol)	Temperature (°C)	Time (min)	Acid Value	ANFIS prediction
13	1	2	65	90	7.01	7.01
14	1.5	1.5	70	80	5.25	5.25
15	2	2.5	65	80	5.26	5.26
16	1.5	1.5	65	90	5.3	5.3
17	1.5	2.5	70	80	5.87	5.87
18	1	1.5	65	80	5.8	5.8
19	1.5	1.5	60	80	4.96	4.96
20	1.5	2	65	80	5.24	5.904
21	1	2	60	80	6.32	6.32
22	1.5	2	65	80	5.23	5.904
23	2	2	65	90	6.22	6.22
24	1.5	2.5	60	80	5.33	5.33
25	1	2	70	80	6.48	6.48
26	1	2.5	65	80	6.48	6.48
27	1.5	2.5	65	70	6.48	6.48
28	1	2	65	70	6.48	6.48
29	1.5	2	65	80	6.48	5.904

The use of particle swarm optimization (PSO), a novel population based search algorithm was used to obtain the actual optimum reaction parameters of Temperature (60°C), reaction time (73 mins), catalyst concentration (1.5) and methanol/oil ratio (1.5 wt%) giving an oil with optimum acid value of 1.488.

3.4. Optimisation of Soybean Soapstock Biodiesel Yield Using ANFIS

3D plots were also used for monitoring parameter interac-

tions for biodiesel yield in ANFIS. From the surface plots, the ANFIS predictions for yield for the 32 runs were made and compared with values obtained from actual runs as shown in Table 10.

Particle swarm optimization (PSO) was also used to obtain the actual optimum reaction parameters of Temperature (54°C), reaction time (42 mins), catalyst concentration (1.5 wt%), stirring speed (300) and methanol/oil ratio (4) to obtain an optimum yield of 99%.

Table 10. Runs for trans esterification of soyabean soap stock using ANFIS.

Run	Reaction Time (min)	Temperature (°C)	Catalyst Concentration (wt%)	Methanol/Oil ratio (mol/mol)	Stirring speed (rpm)	Yield	ANFIS prediction
1	50	50	2	5	400	90.9	93.27
2	50	50	2	7	400	90.48	90.48
3	50	50	3	5	400	91.88	91.88
4	50	50	2	5	400	92.98	93.27
5	65	45	2.5	4	500	77.88	77.88
6	50	45	2.5	4	300	93.08	93.08
7	65	55	2.5	6	500	77.48	77.48

Run	Reaction Time (min)	Temperature (°C)	Catalyst Concentration (wt%)	Methanol/Oil ratio (mol/mol)	Stirring speed (rpm)	Yield	ANFIS prediction
8	50	45	2.5	6	500	93.18	93.18
9	50	45	1.5	6	300	79.38	79.38
10	50	55	2.5	6	300	79.38	79.38
11	50	50	2	5	400	93.78	93.27
12	50	50	2	5	400	91.28	93.27
13	65	55	2.5	4	300	93.98	93.98
14	45	50	2	5	400	78.88	78.88
15	65	55	1.5	4	500	95.08	95.08
16	50	50	2	5	400	95.48	93.27
17	50	50	2	5	600	95.38	95.38
18	50	50	2	5	400	95.18	93.27
19	50	50	1	5	400	79.48	79.48
20	50	55	1.5	4	300	96.8	96.8
21	65	45	2.5	6	300	77.98	77.98
22	50	55	1.5	6	500	78.48	78.48
23	65	45	1.5	6	500	77.58	77.58
24	50	60	2	5	400	78.48	78.48
25	50	50	2	5	200	93.98	93.98
26	50	55	2.5	4	500	93.58	93.58
27	50	45	1.5	4	300	79.48	79.48
28	50	50	2	3	400	93.08	93.08
29	65	55	1.5	6	300	76.48	76.48
30	50	55	1.5	5	400	77.78	77.78
31	50	45	2	5	400	94.98	94.98
32	55	45	2.5	4	500	78.88	78.88

3.5. Comparism of Reaction Parameters

Particle swarm optimization applied to the quadratic model of ANFIS was used in the prediction of acid values from esterification and yields from transesterification of soya soapstock. A comparison of the optimization results obtained initially using RSM and the results obtained using ANFIS were also compared as seen in Tables 11 and 12 to highlight their efficiencies as optimization tools in esterification and transesterification reactions.

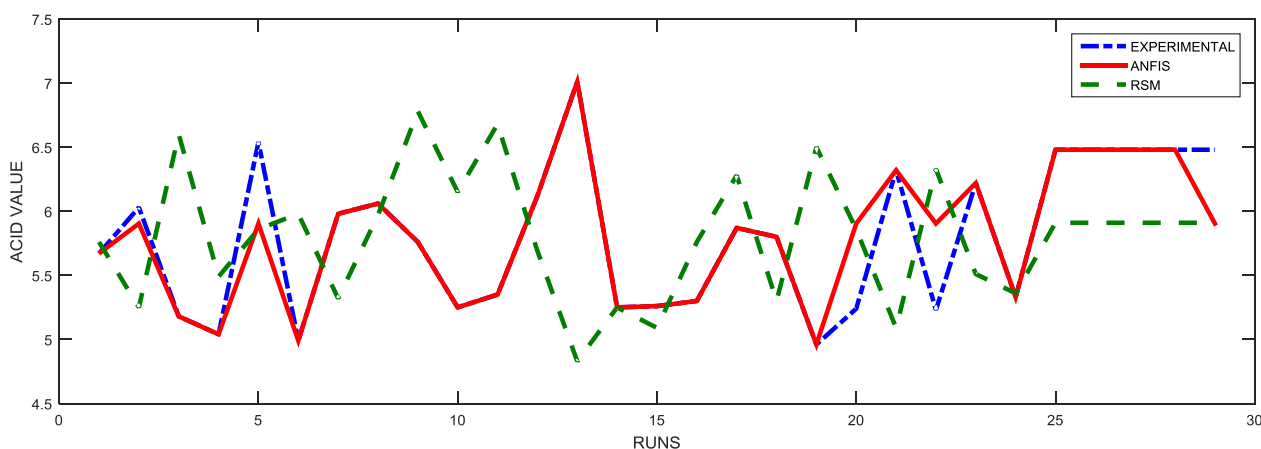
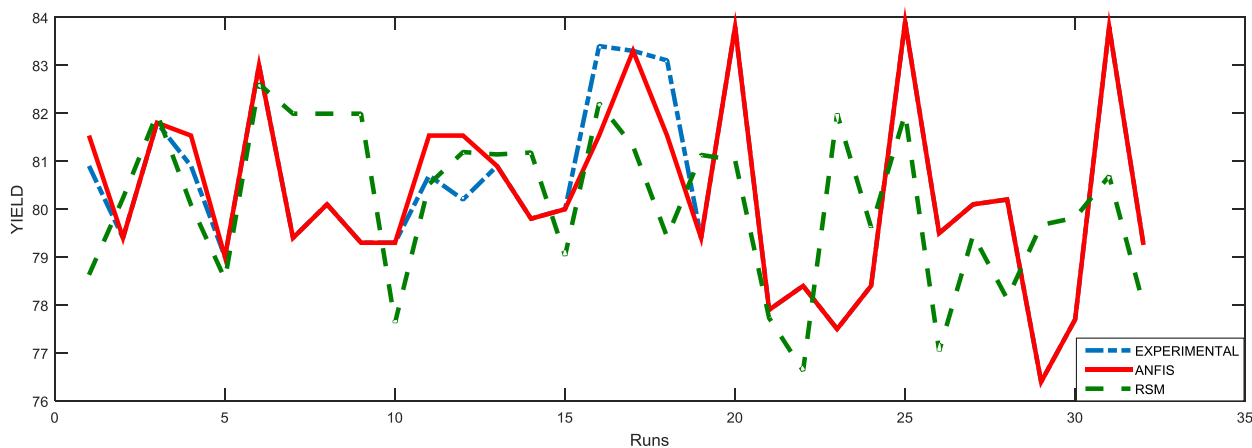
Table 11. Parameters obtained from optimization of esterification of soya soapstock using RSM and ANFIS.

Parameter	RSM	ANFIS
Temperature	61.63	60
Time	74.01	73
Catalyst concentration	1.77	1.5
Methanol/oil ratio	1.52	1.5
Acid value	4.956	1.488

Table 12. Parameters obtained from optimization of transesterification of soya soapstock using RSM and ANFIS.

Parameter	RSM	ANFIS
Temperature	50.68	54
Time	53.36	42
Catalyst concentration	1.77	1.5
Methanol/Oil ratio	4.36	4
Speed	407.6	300
Yield	97.29	99.91

It can be observed that optimization using ANFIS had better yields in transesterification compared to optimization using RSM while lower (optimum) acid values was obtained in the esterification of soya soap stock using ANFIS compared to optimization using RSM. The reaction parameters used to obtain these optimum values were also more favorable in optimization using ANFIS. Plots of optimized values using RSM and ANFIS were compared to ascertain the reliability of both optimization techniques in the 3 processes.

**Figure 5.** Comparison plots for optimization of esterification of soya soapstock.**Figure 6.** Comparison plots for optimization of transesterification of soya soap stock.

It can be observed from the comparison plots using ANFIS and RSM for the optimization of the transesterification of soya soap stock as seen in Figure 6 that optimization using ANFIS mirrors the trend created by the actual showing it is a more precise optimization technique compared to RSM which showed high deviation from the actual and thus can't be said to be a true reflection of the actual. This also applies to the esterification of soya soap stock as seen from Figure 5. Both model response prediction curves tracked the actual response curve to an acceptable extent but ANFIS response curve super-imposed the actual response curve from fifth to twentieth runs for esterification and between twentieth to thirty-second runs in transesterification of soybean soapstock. This implies that ANFIS model had better prediction accuracy when compared to quad-

It can be observed that optimization using ANFIS had better yields in transesterification compared to optimization using RSM while lower (optimum) acid values was obtained in the esterification of soya soap stock using ANFIS compared to optimization using RSM. The reaction parameters used to obtain these optimum values were also more favorable in optimization using ANFIS. Plots of optimized values using RSM and ANFIS were compared to ascertain the reliability of both optimization techniques in the 3 processes.

ratic model of the RSM and should be utilized for further studies for both processes.

ANFIS achieved better prediction and can generally be seen as an efficient optimization technique because of its adaptive and automated nature. The adaptive nature of the system combined neural capabilities to learn the rules and carefully analyse the data with its fuzzy logic inference capabilities. This ensured precise prediction and ability to accommodate problem solving rules that helps in its decision making. The automated nature ensures it can learn from large data to be applied to solving problems. It can thus be concluded that ANFIS is a better optimization technique for the above named processes.

3.6. Error Analysis of Optimized Variables

Error analysis on the 2 optimisation techniques (RSM and ANFIS-PSO) was carried out to highlight their suitability to the esterification and transesterification processes. The error analysis methods used were Residual sum of squares (RSS), Mean absolute error (MAE), Root mean square error (RMSE), Coerrelation coefficient (R), Coefficient of determination (R^2), Adjusted R^2 , Absolute average deviation (AAD) and Mean absolute percent error (MAPE).

It can be observed from Table 13 that RMSE and MAE both recorded smaller values using ANFIS when compared to RSM and thus highlighting ANFIS as a better fit with closer predicted and actual values.

Table 13. Error analysis of the esterification and transesterification of soybean soapstock.

Error analysis method	Esterification of soya soapstock		Transesterification of soya soapstock	
	ANFIS	RSM	ANFIS	RSM
RSS	1.647	21.545	18.453	433
MAE	0.092	0.711	0.29	3.057
RSME	0.238	0.862	0.759	3.678
R	0.909	0.298	0.995	0.899
R^2	0.828	0.089	0.99	0.81
ADJ R^2	0.821	0.044	0.989	0.804
AAD	6.13E-17	0.0027	0.00045	-1
MAPE	1.595	12.355	0.312	3.596

The AAD and MAPE error analysis methods also recorded lower values when using ANFIS. This further reinforces the claim of ANFIS being a better fit than RSM as an optimization technique in esterification/transesterification reactions.

4. Conclusion & Recommendation

4.1. Conclusion

Findings from this work has led to the following conclusions:

- 1) Biodiesel could be obtained from esterification/transesterification of soybean soapstock.
- 2) Improved yield can be obtained from soybean soapstock (a low quality lipid) through a two-step transesterification process and the use of a co-solvent (n-hexane) in the transesterification reaction.
- 3) ANFIS gave higher and more reliable predictions when compared to RSM in esterification and transesterification reactions of soybean soapstock.

tion reactions of soybean soapstock.

4.2. Recommendation

- 1) The use of soapstock from different plant sources should be investigated further as feedstock for biodiesel production due to its reduced use as both animal feed source and production of soap.
- 2) The use of co-solvents in the transesterification of low quality lipids should be encouraged in the search for an economically viable feedstock for biodiesel production.
- 3) Different optimization techniques should be tested and established for different processes/feedstocks as their viability in biodiesel production is relative.

Abbreviations

RSM	Response Surface Methodology
ANFIS	Adaptive Neuro-Fuzzy Inference System
RSS	Residual Sum of Squares

MAE	Mean Absolute Error
RMSE	Root Mean Square Error
R	Coerrelation Coefficient
R ²	Coefficient of Determination
AAD	Absolute Average Deviation
MAPE	Mean Absolute Percent Error

Author Contributions

Chinedu Gabriel Mbah: Conceptualization, Formal Analysis, Methodology, Project administration, Software, Writing – original draft

Francisca Unoma Nwafulugo: Data curation, Investigation, Resources, Supervision, Validation, Visualization

Njideka Ophelia Ezetoh: Writing – original draft, Writing – review & editing

Conflicts of Interest

The authors declare no conflicts of interest.

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