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Modelling and Optimization of Biodiesel Production Process Parameters from Jansa Seed Oil (*Cussonia bateri*) Using Artificial Neural Network

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Abstract: Biodiesel has been referred to as a perfect substitute for diesel fuel due to its numerous promising properties. They are renewable, clean, increases energy security, improves the environment and air quality and also provides some good safety benefits. This study is focused on the investigation of the use of natural heterogeneous catalysts for production of biodiesel from jansa seed oil, as well as the implementation of artificial neural network (ANN) for the prediction of biofuel yield and process parameters. The biodiesel was produced through transesterification reaction by reacting jansa seed oil (FFA) with methanol (alcohol) to yield methyl ester. Waste periwinkle shell was prepared in 3 different forms; raw, calcined and acidified. The percentage yield of the methyl ester obtained were calculated and tabulated. The process parameters considered were methanol-oil mole ratio, catalyst concentration, agitation speed, reaction temperature and reaction time. The results of this research work revealed that the calcined periwinkle shell catalyst produced higher yield of biodiesel, compared to the yield obtained from the raw and acidified catalyzed process. The properties of the fatty acid methyl esters were within the standard range. The experimental and predicted yield were marginally the same. Hence, the model accurately predicted the yield with acceptable coefficient of determination and low mean squared error (MSE). The results demonstrate the flexibility of ANN model and the improvement of the model in terms of performance prediction when solving problems with stochastic dataset, especially the transesterification of biodiesel.

Keywords: Jansa Seed, Catalyst, Transesterification, Artificial Neural Network

1. Introduction

The continuous need for a substitute to diesel has necessitated the quest for a fuel which is non-toxic, biodegradable, environmentally friendly and renewable. This has drawn the attention of developing countries like Nigeria to focus more on problems related to renewable energy [1]. In recent time, biodiesel has gained so much attention because of their high economic value and acceptability. They are dependable alternative source of fuel and are known for their low sulphur content with perfect lubrication property. They are produced from transesterification process between a free fatty acid and a primary alcohol to yield a methyl ester using a suitable catalyst [2]. The four group of catalyst generally used for biodiesel production are base catalysts, acid catalysts, enzymes and nanocatalyst [3]. Acid catalyst requires high molar ratio of the alcohol to oil at increased reaction time and pressure. The metallic materials and engines are susceptible to corrosion as a result of the acid catalyzed transesterification. Favorably, base catalysts do not require long reaction time and have low molar ratio of alcohol to oil, they are relatively cheap with good catalytic activity and catalyst concentration [2]. The low cost and reusability nature of heterogeneous catalyst makes its use ever viable. They do not release large amount of waste water nor undergo saponification rather they require less number of stages for processing [4]. The drawback experienced with the use of enzymatic catalyst is that they are expensive for the industrial scale production of biodiesel [5]. Enzyme catalyzed processes have several benefits when considering conversion, yield and reusability. Lipase mediated transesterification has increased the rate of reaction followed by high conversion, although the activity of free enzyme is reduced as stability is equally reduced. Nanobiocatalyst focuses exclusively on the transesterification of oils using methanol to produce fatty acid methyl esters [6].

Nanocatalyst in current years are the most efficient for heterogeneous catalysis owing to their effectiveness in improving the approach to heterogeneous catalysis. They play a vital role in the general productivity of biodiesel [7] (Avinash *et al*., 2020). Nano catalysts are promising alternatives for efficient production of biodiesel from oils and fats as they have high specific surface area and high catalytic activities eliminating the specific problem of mass transfer resistance associated with conventional catalysts [8-10]. Current research relies on less expensive, abundant feedstocks, novel production and purification technologies for production of biodiesel [11-12].

Cussonia bateri commonly known as Jansa in Cameroun and in Nigeria is a small twisted savanna tree with thick corky bark. The leaves are oborate with lateral nerves; the flowers are greenish white, with whitish fruits and very soft brittle wood. They are abundant in nature and do not compete with the food chain. The seed has a pleasant aroma and becomes oily on storage. This gave rise to the interest to investigate the contents of the plant seed. [13]. Natural heterogeneous catalyst from carbonaceous materials have been reported as a stable and efficient recyclable catalyst for biodiesel production [14-16]. Periwinkle shell which often constitutes environmental pollution due to their poor management; usage of these wastes for solid catalyst preparation can provide cheaper catalysts for biodiesel conversion and as well act as waste disposal strategy [17].

2. Artificial Neural Network (ANN) Model

An artificial neural network (ANN) is a computing system designed to process the way the human brain analyzes and processes information. It is the origin of artificial intelligence (AI) which proffer solutions to lots of problems proven to be difficult or impossible by human or statistical standards. It is a current method of computation which makes use of non-linear modelling and complex dataset [18-23]. It plays a vital role in connecting experimental data and theoretical facts in various fields. It comprises of an input, hidden and output layer. The modeling has a high potential to contribute to the development of renewable energy systems by accelerating biodiesel research [24]. The experimental design indicates how to obtain the optimal response. [2] (Ayoola *et al*., 2019). The ability to detect accurately from minute to large data sets, relatively cheap with less time consumption has made the use of ANN more popular. They are quite adaptable with numerous disciplines like neuroscience, various computation analysis, chemical, environmental engineering, engineering design and applications [25.]. The aim of this research is to evaluate the performance of different prepared samples of periwinkle shell as a catalysts with regards to their yields of biodiesel in the transesterification process of jansa seed oil and also to formulate a system that relates the process variables and the yield of biodiesel using ANN modelling technique.

3. Materials and Method

3.1. Materials and Reagents Used

The materials used for this experiment includes: Beaker, stirrer, conical flask, fume cupboard, magnetic stirrer, density bottle, pensky martens flash point tester, pour point tester, powdered seed samples (oil, esters, ester/ diesel blends and diesel), separatory funnel, soxhlet extractor, calcinator, muffle furnace, oven, digital magnetic heater, digital weighing balance, TECHNO R175A diesel engine, FTIR Spectrophotometer (8400SSHIMADZU), Emission Scanning Electron Microscope (JSM-670IF), GC- MS QP2010 Plus Shimadzu Japan, X-ray Fluorescence. Correspondingly, the reagents used for this experiment includes: Potassium hydroxide (BDH), hydrochloric acid (Sigma-Aldrich), concentrated sulphuric acid (Sigma-Aldrich), chloroform (BDH), distilled water, methanol, ethanol, glacial acetic acid (BDH), iodine monochloride, methanol (BDH), n-hexane (BDH), phenolphthalein, indicator, potassium iodide, sodium hydroxide, sodium thiosulphate, sodium chloride, hydrochloric acid, tetraoxosulphate (vi) all of analytical grade.

3.2. Sample Collection and Oil Extraction

The method adopted in the study used raw oil seed of *Cussonia Bateri,* which was purchased from local retailer in Ogbete main market, in Enugu State. The sample was identified by a taxonomist and was sun dried and ground using industrial blender. It was subsequently sieved using sieve size 500µm.

3.3. Oil Extraction

Oil was extracted from the sample by soxhlet extraction. Sample was weighed into a semi-permeable cotton material and placed into the thimble of a 500ml soxhlet extractor. 400ml n-hexane was measured into a 500ml flat bottom round flask. The soxhlet with the extraction thimble containing the sample in a semi-permeable membrane was connected with the condenser which were fitted to the flat bottom flask containing n-hexane. The soxhlet extraction system was heated on a hot plate while water was allowed to circulate at the outer jacket of the condenser. The extraction was discontinued when oil was completely extracted from the sample. The de-fatted sample in the semi permeable membrane was discarded, while the oil and n-hexane mixture in the flat bottom flask were separated by distillation. During distillation, the n-hexane were distilled over while oil remained in the flask.

3.4. Catalyst Preparation and Characterization

The first sample of the Periwinkle shell was obtained by the sea side and the second sample was purchased from a popular market in Enugu state. It was washed, dried and ground to fine paste. Some portion of the PSA was calcined up to a temperature of 800°C while 10g each of the PSA was acidified with conc. sulphuric acid (10ml). It was washed repeatedly till all traces of the acid have been washed off. They were packaged and sent for further analysis using Scan Electron Microscope and Fourier Transform Infra-red.

3.5. Biodiesel Production (Transesterification)

The transesterification reaction to produce methyl ester was carried out in a 500 ml, round bottom, glass, spherical, three neck reactor. Calculated amount of methanol and modified PSA catalyst was added in the amounts established for the experiment and pre-stirred for 10 minutes for proper dissolution after which 50ml of oil was added. A reflux condenser with cold water circulating at the outer jacket was fitted to mid neck of the reactor. Mercury in glass thermometer held in plastic bung was fitted to the right neck of the reactor. A capsular stirring nob was placed into the reactor through the left neck of the reactor and closed using a plastic bung. The setup was placed on a magnetic heating mantle and switched on. The stirring system was also switched and maintained at an established speed and regulated at required temperature, taking this moment as time zero of the reaction. Each reaction was allowed to last for the required time at specific temperature. After the transesterification reaction, the product was allowed to stand for twelve hours in a separating funnel for glycerol separation. The crude glycerol was removed through the funnel tap leaving the methyl esther, (biodiesel) behind. The biodiesel was washed with hot water and dispensed into a 250 ml beaker. It was heated at 105°C to remove water molecules from the biodiesel. The biodiesel was allowed to cool and stored in calibrated specimen bottles.

4. Experimental Design

Optimum reaction conditions were determined as methanol oil molar ratio, speed of agitation, catalyst concentration with quantity and reaction time were obtained. The following parameters for enhancing the% FAME yield were optimized. Totally, twenty five sample data were required for this experiment. The dependent variables for this study were% FAME yield and the independent variables selected were: methanol/ oil molar ratio, catalyst amount, speed of agitation and temperature. The experimental data of the FAME yield for various catalyst amounts, speed of agitation, molar ratio, and temperature are presented in Table 1. Dataset are fed into ANN toolbox from MATLAB, for the kinetic modeling of the process. The properties of the jansa seed oil FAME produced were also measured and compared with the study of Chaudbery [26], as presented in Table 2. In line with the study of [27] Caniro *et al*., (2010), the properties of the FAME were impressive and within limit with the exception of kinematic viscosity, acid value and calorific value. Therefore, this biodiesel may be used for blending with other fuels.

4.1. Effect of Process Parameters (Periwinkle)

Experimental results to illustrate the FAME yield obtained from the 3 forms of PSA.

Temperature = 55°C, Mole Ratio = 10:1, Time = 3 hrs, Speed= 300rpm.

Table 1. *Table Representing Input Variables and Experimental Results.*

Std Run	A:A	Input variable Catalyst. Conc. % wt of Oil	Exp. Value Yield 1 vol.% (Raw)	Exp. Value Yield 2 (vol.%) (acid activated)	Exp. Value Yield 3 (vol.%) (calcined)
1	9	0.75	60	69	73
2	6	3.75	60	69	73
3	2	0.75	64	69	73
4	14	3.75	64	69	73
5	10	0.75	60	74	73
6	8	3.75	60	74	73
7	4	0.75	64	74	73
8	19	3.75	64	74	73
9	1	0.75	60	69	76
10	13	3.75	60	69	76
11	23	0.75	64	69	76
12	11	3.75	64	69	76
13	12	0.75	60	74	76
14	24	3.75	60	74	76
15	18	0.75	64	74	76
16	3	3.75	64	74	76
17	7	-0.75	62	71.5	74.5
18	20	5.25	62	71.5	74.5
19	15	2.25	58	71.5	74.5
20	5	2.25	66	71.5	74.5
21	25	2.25	62	66.5	74.5

Std Run	A:A	Input variable Catalyst. Conc. % wt of Oil	Exp. Value Yield 1 vol.% (Raw)	Exp. Value Yield 2 (vol.%) (acid activated)	Exp. Value Yield 3 (vol.%) (calcined)
22	17	2.25	62	76.5	74.5
23	21	2.25	62	71.5	71.5
24	22	2.25	62	71.5	77.5
25	16	2.25	62	71.5	74.5

Table 2. *Properties of FAME produced from Jansa oil.*

S.no	Property	Values from the experiment	Standard value/range Canoira et al. [36]
1.	Kinematic viscosity (mm^2 /s a;t 40 C)	17.77	3.5–5.00
2.	Density (kg/m^3)	892	860–900
3.	Acid Value (mg KOH/g)	1.4	<0.50
4.	Flash point (C)	254	>120
5.	Water content (%)	0.009	<0.0005 or\500 mg/kg
6.	Calorific value (MJ/kg)	29.69	37.27–38.22
7.	Oxidation stability at 110 C (h)	11	>6.0
8.	Sulfur content (mg/kg)	0.5	<10

4.2. ANN Model Validation

The ANN model was conducted using the experimental data set obtained from the lab reactor, as presented in Table 1. This was implemented using MATLAB 2020 version. MATLAB Neural network toolbox was employed. The ANN model with three-layered feed forward (FF) back propagation (BP) network was used and the architecture of the ANN network model was presented in Figure 2. The system architecture has five (5) input layer, twelve (12) hidden layer and three (3) output layers. The simulation process for obtaining good result is presented in Figure 1.

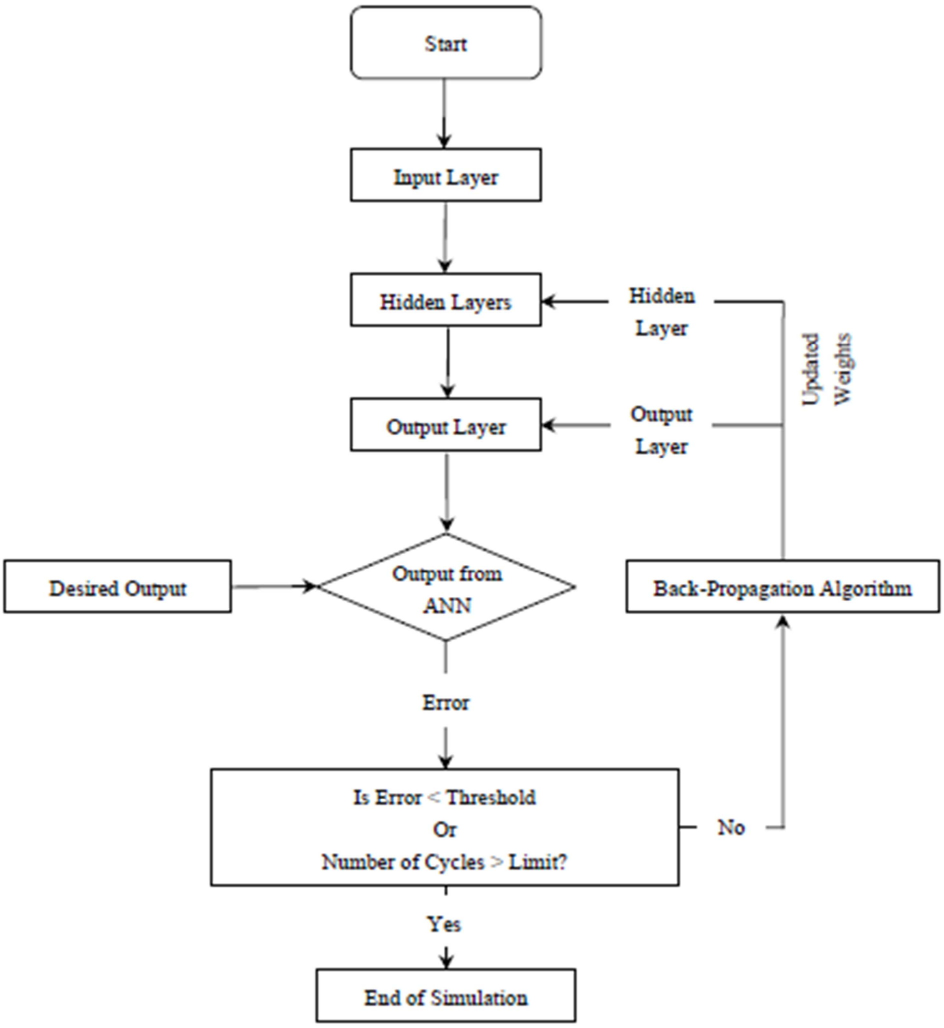

Figure 1. *ANN Flow chart showing the Simulation Process.*

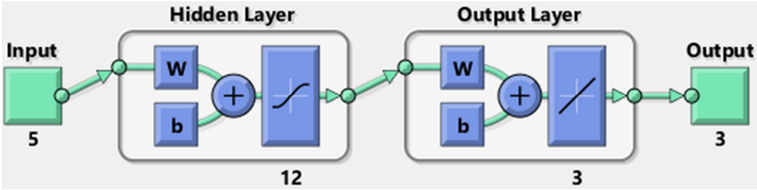

Figure 2. *Architecture of the ANN Network.*

Iteration was performed several times and feasible solutions were obtained showing good prediction of the model in terms of output of the process, which yielded a good approximation for the jansa oil transesterification process. From the simulation result, fifty (50) iteration process was observed on the ANN toolbox. The result obtained confirms the efficiency of the ANN model with low MSE and R value at the training stage, the validation and testing confirm good R values. The result of Table 3 shows that ANN is efficient for predicting the FAME yield at conditions within the range of considered variables.

Table 3. *Best ANN Result Obtained from Experiment.*

Results			
	Samples	MSE	R
Training	17	3.14575	0.943775
Validation	4	8.69509	0.903144
Testing	4	6.57467	0.947909

Presented in Figure 3, is the error histogram for the entire process, showing clearly the training, validation, testing and negligible error. The error histogram presented has the training set presented in blue lines, the testing dataset in red and the validation dataset in green font. The point where the error tend to zero is shown on the graph on a thin straight line in orange font. The algorithm used in this context is the back-propagation algorithm.

Figure 3. *Error Histogram of the Model.*

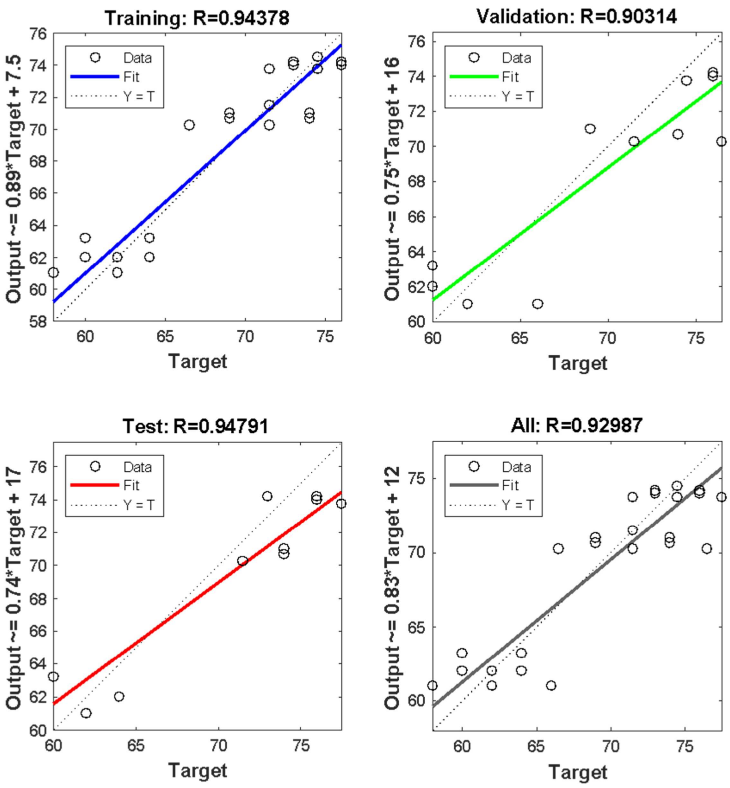

Figure 4. *ANN Regression Plot for Training, Testing and Validation.*

As earlier stated, the Figure 4 reveal the plots of regression for training, validation, test and overall plot, using the result generated from the experiment. R denotes the correlation coefficient, which indicates the degree of association between the variables in question. It is equally important to state the limit of values of the correlation coefficient with ranges from zero (0) to one (1). The zero value indicates no linear relationship, while the value of one (1) indicates that the obtained variables are perfectly related. This further means that the solution obtained is adequate when the coefficient of determination falls between the range of 0.7 and 1.0. A value of less than 0.7 is considered to be below the range of acceptable solutions [28].

The best validation performance in terms of biodiesel yield was obtained at epoch 3. The satisfactory point is the point where there is tendency of obtaining perfect connection between determined variables. The MSE and R value obtained is presented in Table 2 and the final predicted result obtained is available in Table 4. Figures 2-4 demonstrate the relationship between the experimental and predicted result.

From Table 2 and Figure 4, it is a clear indication that the MSE value obtained is significantly low while the R values obtained for training, testing and validation are 0.944, 0.903 and 0.948 respectively. The overall value of 0.92987 was obtained as shown in Figure 4. The final outcome of the training shows clearly a worthy agreement between the predicted ANN solution model and the result obtained from experimental yield of biodiesel. The error is significantly minimal. Analysis of result is presented in Table 4.

Table 4. *ANN Solution Table for Predicted Values of Raw, Acid Activated and Calcined.*

Std Run	A:A	Input variable	Output value	Output Value	Output value	ANN Predicted value	ANN Predicted value	ANN Predicted value
		Catalyst. Conc.	Yield 1	Yield 2	Yield 3	Yield	Yield	Yield
		% wt of Oil	vol.% (Raw)	(vol.%) (acid activated)	(vol.%) (calcined)	vol.% (Raw)	(vol.%) (acid activated)	(vol.%) (calcined)
1	9	0.75	62.0000	70.6667	74.0000	62.0000	70.6667	74.0000
2	6	3.75	63.2000	71.0000	74.2000	63.2000	71.0000	74.2000
3	2	0.75	62.0000	70.6667	74.0000	62.0000	70.6667	74.0000
4	14	3.75	63.2000	71.0000	74.2000	63.2000	71.0000	74.2000
5	10	0.75	62.0000	70.6667	74.0000	62.0000	70.6667	74.0000
6	8	3.75	63.2000	71.0000	74.2000	63.2000	71.0000	74.2000
7	4	0.75	62.0000	70.6667	74.0000	62.0000	70.6667	74.0000
8	19	3.75	63.2000	71.0000	74.2000	63.2000	71.0000	74.2000
9	1	0.75	62.0000	70.6667	74.0000	62.0000	70.6667	74.0000
10	13	3.75	63.2000	71.0000	74.2000	63.2000	71.0000	74.2000
11	23	0.75	62.0000	70.6667	74.0000	62.0000	70.6667	74.0000
12	11	3.75	63.2000	71.0000	74.2000	63.2000	71.0000	74.2000
13	12	0.75	62.0000	70.6667	74.0000	62.0000	70.6667	74.0000
14	24	3.75	63.2000	71.0000	74.2000	63.2000	71.0000	74.2000
15	18	0.75	62.0000	70.6667	74.0000	62.0000	70.6667	74.0000
16	3	3.75	63.2000	71.0000	74.2000	63.2000	71.0000	74.2000
17	7	-0.75	62.0000	71.5000	74.5000	62.0000	71.5000	74.5000
18	20	5.25	62.0000	71.5000	74.5000	62.0000	71.5000	74.5000
19	15	2.25	61.0000	70.2500	73.7500	61.0000	70.2500	73.7500
20	5	2.25	61.0000	70.2500	73.7500	61.0000	70.2500	73.7500
21	25	2.25	61.000	70.2500	73.7500	61.000	70.2500	73.7500
22	17	2.25	61.0000	70.2500	73.7500	61.0000	70.2500	73.7500
23	21	2.25	61.0000	70.2500	73.7500	61.0000	70.2500	73.7500
24	22	2.25	61.0000	70.2500	73.7500	61.0000	70.2500	73.7500
25	16	2.25	61.0000	70.2500	73.7500	61.0000	70.2500	73.7500

4.3. Further Discussion on Biodiesel Prediction Model Using ANN Technique

As indicated in Table 2, which represent sixty eight (68) percent of obtained dataset has been sectioned for training, and the remaining thirty two percent are sectioned in such a way that sixteen (16) percent was used for testing and another sixteen (16) percent for validation in order to obtain a realistic outcome. The value obtained after the first process simulation demonstrates a high mean squared error (MSE) value and a low coefficient of determination (R) value. Continuous simulation process was conducted until a satisfactory result was obtained from the neural fitting system of the ANN toolbox.

The predictions were made using the developed ANN model at the conditions (catalyst =0.75-3.75, temperature = 55°C, time =3 hrs, mole ratio= 10:1 agitation speed=300rpm) to study the effect of molar ratio on FAME yield.. It was considered appropriate to develop a kinetic model at optimized conditions. The results obtained for these conditions from the ANN model have been used to develop the kinetic model. The kinetic model is in general reversible reaction model, in which both forward and backward reactions are second order, first order with respect to each of the reactants and products [29-31].

5. Conclusion

The research evaluated the performance of biodiesel production in the transesterification process of jansa seed oil catalyzed with PSA in 3 different forms; raw, acidified and catalyzed. The result of the research revealed that PSA calcined produced higher yield of biodiesel as seen in tables 1, 2 and 3. Calcination which is a form of purification removes volatile substances and renders them friable. Concentration of the catalyst increased resulting to greater amount of available basic sites which are the active basic phase in the transesterification. This led to higher conversions of biodiesel. Higher values of correlation coefficient (0.93) testify the goodness of fit of the kinetic model. The results showed the efficacy of ANN in giving solution to cumbersome problems experienced in the transesterification of biodiesel. The observations of Figures 1-4 have elucidated that ANN is a superior research tool in data modelling of jansa seed oil biodiesel transesterification. The ANN back-propagation model produced smaller deviations with high level grander solution with the coefficient of determination obtained at minimized error. ANN provided the accurate analysis of the complex problems. In this way it would be possible to conduct time and cost efficient studies instead of expensive and long experimental study.

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