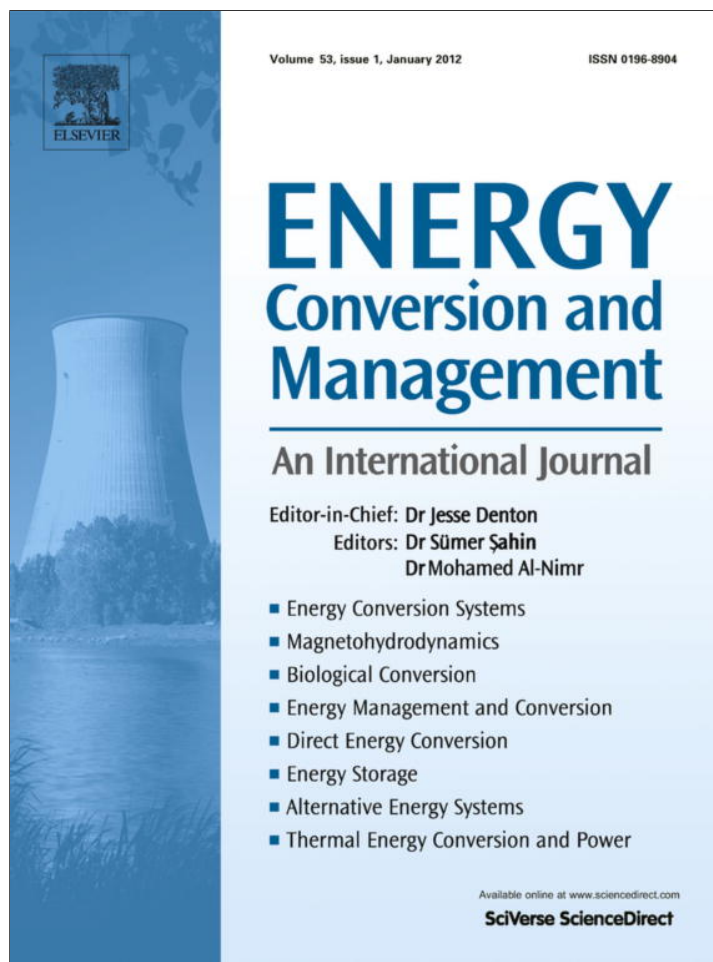


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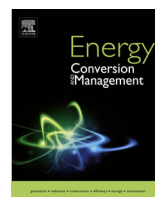
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Effect of reaction temperature and time on neem methyl ester yield in a batch reactor

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ABSTRACT

Experimental investigation of neem methyl ester yield in a batch reactor at different process conditions of temperature and reaction duration was carried out using a 2-factor, 5-level full factorial experimental design. Reaction temperature was varied between 40 °C and 60 °C, while reaction time was studied in the range of 30–120 min. The study, which was carried out using 1% w/w catalyst amount and alcohol to oil ratio of 6:1, showed that reaction temperature had a highly significant effect ($p < 0.01$) in comparison with reaction time, which had a significant effect ($p < 0.05$) on methyl ester yield. Six (6) empirical models were developed for the response variable of methyl ester yield using a non-linear regression analysis method, facilitated by NLREG version 6.3 software. Results showed that the mean predicted ester yield values and mean experimental values were not statistically different at the 95% confidence level, whereas the maximum deviation observed was 6.9%.

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1. Introduction

Global carbon dioxide emission is rising and has already exceeded the dangerously high level of 450 ppm [1]. This has led to serious concerns regarding on-going and impending climate change phenomena in many parts of the world, necessitating more efforts towards reducing the use of fossil fuels by increasing the application of renewable energy derived fuels, especially for transportation. Biofuels, particularly those derived from second generation biomass resources have been shown to be useful alternatives to conventional transport fuels. Biodiesel – a first generation biofuel – derived from vegetable oils and fats is a suitable alternative fuel for compression ignition engines. As an alternative fuel, biodiesel is becoming increasingly important due to diminishing petroleum reserves and adverse environmental consequences of exhaust gases from petroleum-fuelled engines [2]. The biodiesel manufacturing process converts oils and fats into mono alkyl esters, or biodiesel. These chemicals are also referred to as fatty acid methyl esters (FAMES).

Although straight vegetable oils (SVOs) have been applied to compression ignition (CI) engines as alternative fuels, starting with Rudolph Diesel's successful trials in the early 19th Century, when he used peanut oil to power his CI engine, research has shown that raw vegetable oils can cause long-term problems to the engine

owing to their high viscosity [3,4]. Among other methods of conversion [5], transesterification of vegetable oils into biodiesel has become a standard way of producing fuel grade products from them by reducing their viscosity to acceptable values of between 1.9 and 5.9 mm²/s. Biodiesel has numerous advantages over fossil-derived diesel fuels as reported elsewhere [3,4,6], but the major factor limiting its widespread utilization is high cost of production. The high production cost of biodiesel derives directly from the high price of vegetable oil raw materials, most of which are edible (first generation biofuel sources) and serve as food for man. Non-edible oils such as neem, jatropha and other oils are, therefore, becoming more important as biodiesel feedstock [2].

Apart from high vegetable oil prices, the processing cost of biodiesel is also high, constituting about 20–25% of total cost of production in the acid/base catalyzed methyl ester process. Heterogeneous catalysts are becoming increasingly important [7], but homogeneous catalysts have some advantages in large scale biodiesel production [8]. There is a large number of commercial plants producing biodiesel by transesterification based on base catalyzed (caustic) homogeneous transesterification of vegetable oils due to a number of advantages of such catalysts [7,9]. Modelling the ester yield as a function of process parameters could enable better estimation of biodiesel yield from the transesterification process and ultimately reduce the number of practical experiments required for process optimization and therefore, the production cost of biodiesel. Some process parameters that affect the ester yield during transesterification of vegetable oils include alcohol-to-oil ratio,

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catalyst amount (which varies between 0.5–1.5%), reaction duration and temperature. Other factors, such as degree of agitation, and nature of feedstock are also of importance [10].

Neem tree (*Azadirachta indica*) thrives very well in Nigeria and has indeed been applied as a desert control agent in parts of northern Nigeria and neighbouring West African countries. The tree grows naturally in areas with 450–1200 mm annual rainfall but has been introduced successfully even in areas where the rainfall is as low as 150–250 mm. Neem grows on altitudes up to 1500 m. It can grow well in wide temperature range of 0–49 °C and requires a lot of sunshine [11]. However, it cannot withstand water-logged areas and poorly drained soils. The pH range for the growth of neem tree lies in between 4 and 10. Neem trees have the ability to neutralize acidic soils by a unique property of calcium mining [12]. The oil is relatively abundant in Africa and portends great prospects for the development of agriculture. In spite of these prospects, there are few reports on the energy applications of neem oil, in comparison with other non-edible oil crops like jatropha.

The present research work is intended to contribute towards better understanding of the biodiesel process with neem oil feedstock. It dwells on the experimental investigation and empirical modelling of the influence of reaction temperature and duration on neem oil methyl ester yield at 1% catalyst amount and a 6:1 alcohol to oil ratio, using non-linear regression analysis. The ranges of reaction temperature and duration studied were chosen based on literature reports [13].

2. Materials and methods

The fatty acid profile of the neem oil was first studied using Agilent 7890A Series Gas Chromatograph with split/splitless injector, Agilent Technologies (Stevens Creek Blvd., Santa Clara, CA, United States). Results of the chromatographic analysis, presented in Table 1, indicate that the molecular mass of the oil is 871.992. It also showed that the neem oil feedstock was about 99% pure.

The reactor used to carry out the experiments is a 50 L capacity batch reactor (Fig. 1) designed and fabricated for the study with a cylindrical section (37 cm diameter and 48 cm height), which tapers into a lower conical section for ease of product evacuation [3]. The reactor was constructed using stainless steel plate of 2 mm thickness and lagged with glass wool insulator, whereas a simple flat bar of cast iron was used to fabricate the agitator paddle. The heat supply source was a 2.5 kW electrical coil, controlled by a linear thermoregulator based on LM 35 integrated circuit sensor, which was developed for the study. The thermoregulator was calibrated in neem oil environment against the readings of a K-type thermocouple, BK Precision (Made in Taiwan). The top-entering agitator for the reactor was driven by a 746 W (1 Hp) single phase electric motor, mounted on the reactor stand and supported on a ball bearing to reduce wobbling effect. Agitation speed was maintained at 1350 rev/min or 2.5 rev./s for all the experiments.

Table 1
Fatty acid profile of neem oil. Source: [10]

S. no.	Fatty acid description	Fatty acid name	Composition (%)	^a Range of composition (%)
1	C16:0	Palmitic acid	16.65	16–33
2	C16:1	Palmitoleic acid	0.24	Trace
3	C18:0	Stearic acid	18.39	9–24
4	C18:1	Oleic acid	46.65	25–54
5	C18:2	Linoleic acid	15.41	6–16
6	C18:3	Linolenic acid	0.56	Trace
7	C20:0	Arachidic acid	1.04	Trace
		Total	98.94	

^a Molecular weight = 871.992.

Neem oil used for the present work was purchased from a farm in Katsina State of Nigeria and pretreated via degumming and acid esterification following a procedure described by Ramadhas et al. [14]. 20 L of the pretreated neem oil with water content of 0.20% and free fatty acid (FFA) content of 0.13% was measured out and transferred into the reactor. The thermoregulator sensor was inserted into the reactor, while the agitator was connected to a power supply source.

3. Experimental

The pre-determined quantity of NaOH, corresponding to 1% w/w of the oil was mixed separately with the methanol (in a 6:1 alcohol to oil ratio) and stirred to dissolve it. Although some researchers favour the use of 1.5% catalysts amount in the base catalyzed process [15], others such as Vincente et al. [10] have reported nearly 100% biodiesel yield while using 1% catalyst. The reactor content was first preheated to the desired temperature (at which the thermoregulator was set) according to the experimental plan. When the neem oil had attained the desired temperature and the catalyst completely dissolved, the methanol/NaOH mixture was poured into the oil and the reactor closed tightly. At the end of each reaction, the content of the reactor was transferred into a tall glass cylinder for gravity separation of the glycerol and biodiesel. The supernatant phase (FAME) was carefully separated and washed several times with tap water until a neutral pH value (of 7.0) was attained. The quantity (volumetric yield) of the FAME was measured and converted to percentage yield with respect to the oil. A summary of ester yields (EY) obtained for different treatment combinations (experimental points) is presented in Table 2.

The experimental plan applied in this study is a 2-factor, 5-level factorial design. The factors are reaction duration (time) and temperature. Reaction duration was varied between 30 min and 120 min with a step of 22.5 min, while temperature was studied in the range of 40–60 °C, with a step of 5 °C. To ensure accurate measurement of reaction temperature, a linear thermoregulator was developed based on the LM35 integrated circuit (as described by Anyanwu et al. [3]) and used in this work. A total of 25 treatment combinations were studied, at which transesterification reaction was carried out (Table 2).

To determine the quantity of FAME after each experiment, FAME sample was derivatised using t-butyldimethylchlorosilane. A 1% heptadecanoic methyl ester (HME) in hexane was prepared and HME equivalent to 5% of the total FAME was added to each FAME dilution as internal standard. This mixture was analyzed using Agilent 7890 GC equipped with FID Detector, Agilent 7683 series Injector and Agilent 19091N Column of length 30 m and internal diameter 0.32 mm.

The experimentally determined values of ester yield were presented graphically to enhance understanding of the nature of the curves and, thus facilitate the regression modelling (curve fitting) aspects of the work. Thereafter, NLRG version 6.3 software was used to develop regression models expressing the dependence of

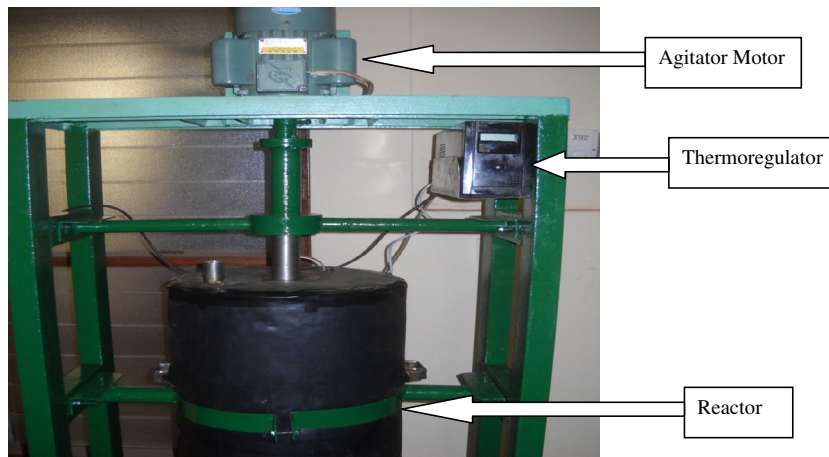


Fig. 1. Front view of the biodiesel reactor.

Table 2

Summary of ester yield (%) for the 5² full factorial experiments.

Temp. (°C)	Time (min)				
	30.0	52.5	75.0	97.5	120.0
40.0	45	55	62	65	80
45.0	58	58	68	63	70
50.0	60	80	85	90	75
55.0	62	78	84	83	78
60.0	75	65	70	60	0 ^a

^a Reaction produced negligible quantity of biodiesel inside a mass of soap.

ester yield on reaction time at each temperature level studied. Using statistical principles, the effects of the reaction parameters studied on the response variable were also quantified and compared.

4. Results

A summary of the results of ester yield for different treatment combinations (experimental points) is presented graphically in Fig. 2. From the plots, the dependence of ester yield on reaction

duration for most of the temperature levels between 40 °C and 60 °C appear to fit well into quadratic curves. The graph shows that the highest ester yields were generally obtained at a reaction temperature of 50 °C, whereas the lowest were at 40 °C and 60 °C.

4.1. Main effects of the reaction temperature and time

In order to properly analyze the effects of the factors (reaction parameters), it is necessary to obtain the average ester yields for each level of time (θ) and temperature (T). For instance,

$$\begin{aligned} \text{Average EY for the lowest level of Time(30 min)} &= \frac{\sum_1^5 \text{EY}(\theta 30)}{5} \\ &= \frac{300}{5} = 60.0\% \end{aligned}$$

For the highest level of Time (120 mins), average EY

$$= \frac{\sum_1^5 \text{EY}(\theta 120)}{5} = \frac{303}{5} = 60.6\%$$

The average values of the EY for each of the five temperature levels were calculated in a similar way:

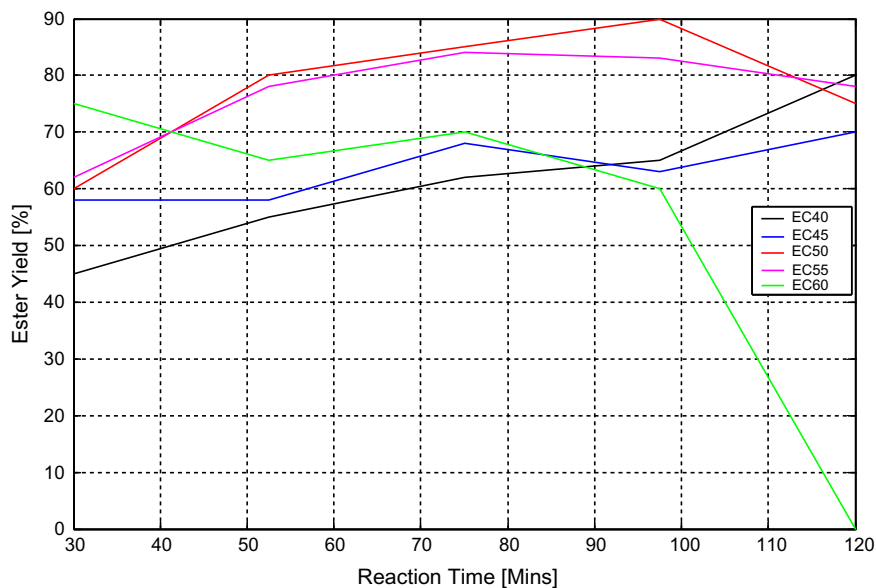


Fig. 2. Graph of ester yield at different treatment combinations.

$$\text{Average EY for } 40\text{ }^{\circ}\text{C} = \frac{\sum_1^5 \text{EY}(T40)}{5} = \frac{307}{5} = 61.4\%$$

$$\text{Average EY for } 60\text{ }^{\circ}\text{C} = \frac{\sum_1^5 \text{EY}(T60)}{5} = \frac{270}{5} = 54.0\%$$

The average values obtained for each factor level are presented in Table 3.

The main effects of each factor on EY is the difference between the average EY for each level and the grand mean of 66.76%. These are presented in Fig. 3, which shows that the effects of both temperature and reaction time were increasing between the first and third levels, but decreasing between the third and fifth levels. However, the effect of temperature appears to be slightly more pronounced than that of time, which is characterized by a squatter curve. Indeed ANOVA shows that the effect of temperature is highly significant ($p < 0.01$), while that of time is significant ($p < 0.05$).

4.2. Interaction effects

An interaction is said to occur when one factor effects the results differently depending on the second factor. In the present set-up, which is a multi-level factorial experiment involving two factors, interaction can be detected by comparing the trend in ester yield values obtained between the lowest and the highest levels of one of the factors. If the trends are easily represented by two parallel lines, this means there was no interaction, but if the lines are not parallel, interaction is said to have taken place. Table 4 shows the trends in EY for different levels of temperature and time. This is clearer as presented in Fig. 4, which shows that there was interaction between reaction temperature and time during the transesterification reactions considering the fact that the two lines are not parallel. ANOVA showed that the effect of factor interaction is significant ($p < 0.05$).

4.3. Modelling in NLREG environment

Ester yield data obtained for the experimental points (treatment combinations) were recorded and used to develop empirical models using a non-linear regression analysis method facilitated by NLREG version 6.3 Software. A number of authors have reported the optimization of methyl ester yield of vegetable oils and fats using response surface methodology (RSM) [16–19], but the use of conventional non-linear regression offers an opportunity to fine-tune the values of the parameters. NLREG is a powerful statistical analysis program that performs linear and non-linear regression analyses, surface and curve fitting [20]. NLREG has been validated using the Statistical Reference Datasets developed by the National Institute of Standards and Technology, USA. NLREG determines the values of parameters for an equation (whose form is specified algebraically by the user), that cause the equation to best fit a set of data values. It can handle linear, polynomial, exponential, logistic, periodic, and general nonlinear functions [20].

NLREG features a full programming language (with syntax similar to C language) for specifying the function that is to be fitted to

Table 3
Average EY values (%) for different levels of temperature and time.

	Temperature averages	Time averages
Level 1	61.4	60.0
Level 2	63.4	67.2
Level 3	78.0	73.8
Level 4	77.0	72.2
Level 5	54.0	60.6

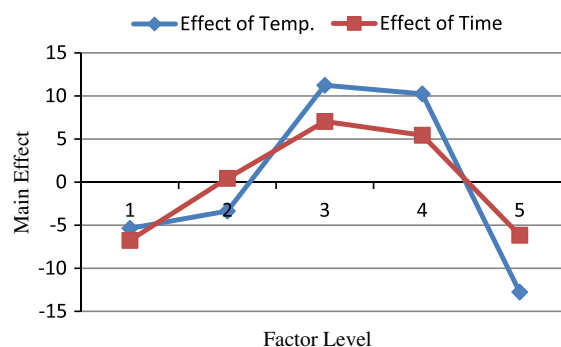


Fig. 3. Main effects of temperature and time on EY.

Table 4
Trends in EY for different Levels of temperature and time.

Level	Temp. ($^{\circ}\text{C}$)	EY (%) at 30 min	Time (min)	EY (%) at 40 $^{\circ}\text{C}$
1	40	45	30	45
2	45	58	52.5	55
3	50	60	75	62
4	55	62	97.5	65
5	60	75	120	80

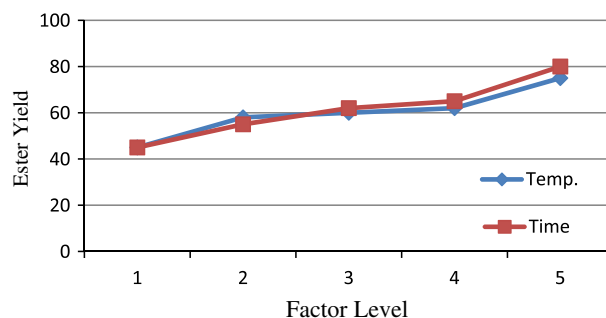


Fig. 4. Trends in EY values between 40 $^{\circ}\text{C}$ and 60 $^{\circ}\text{C}$.

the data. With NLREG it is easy to construct piecewise functions that change form over different domains. Since the NLREG language includes arrays, it is possible to use tabular look-up methods to define the function. The first lines in NLREG are used to declare/introduce the variables and parameters as well as the function. The next few lines are for any other instructions such as “plot” etc., followed by data entry. NLREG performs true non-linear regression analysis and curve fitting, it does not transform the function into a linear form.

After running the program, the analysis generated was evaluated using the coefficient of multiple determination R^2 , Prob t and other statistical indices. This process was repeated with a number of curves until the best combination of the coefficient of multiple determination, Prob t , etc. were obtained. The values of the coefficients generated by NLREG were then adopted in the empirical model.

Among all the curves explored, a quadratic expression was found to have the best fit on the ester yield data obtained experimentally. This conclusion was arrived at considering the high (close to 1.0) values of the coefficient of multiple determination, R^2 obtained in virtually all the cases, coupled with other indicators (e.g. low Prob t).

A quadratic model was developed, expressing the relationship between reaction time and ester yield at 40 $^{\circ}\text{C}$:

$$\text{EY} = 38.64 + 0.00085\theta^2 + 0.2286\theta \quad (R^2 = 92.02\%) \quad (1)$$

where EY is the ester yield (%); and θ is reaction time (min).

For 45 °C, the model is expressed by

$$EY = 53.08 - 0.00014\theta^2 + 0.1501\theta \quad (R^2 = 68.32\%) \quad (2)$$

Similarly, an NLREG model (Eq. (3)) was developed for estimating the ester yield at 50 °C:

$$EY = 19.11 - 0.00988\theta^2 + 1.6592\theta \quad (R^2 = 96.23\%) \quad (3)$$

where both variables have the same meanings as in Eqs. (1) and (2).

The best NLREG curves (models) developed for estimating ester yield at 55 °C and 60 °C are expressed by Eqs. (4) and (5), respectively:

$$EY = 32.78 - 0.0069\theta^2 + 1.2015\theta \quad (R^2 = 98.85\%) \quad (4)$$

$$EY = 30.83 - 0.0162\theta^2 + 1.7450\theta \quad (R^2 = 88.78\%) \quad (5)$$

The developed empirical models (Eqs. (1)–(5)), were used to calculate methyl ester yields at the same treatment combinations, at which the experiments were carried out. Obtained results were then compared with the actual experimental values as presented in Table 5. Except in the case of reactions conducted at 60 °C, all other ester yield values estimated by NLREG models were within $\pm 7\%$ of their experimentally obtained values. Indeed, the maximum deviations for the models were 6.2%, –6.8%, 3.5%, 1.5% and 19.5% at 40 °C, 45 °C, 50 °C, 55 °C and 60 °C; whereas the average deviations were 0.20%, 0.18%, 0.04%, 0.04% and –2.12 respectively. Deviations were calculated according to the following equation:

$$\text{Deviation (\%)} = \frac{100 (\text{Calculated yield} - \text{Experimental yield})}{\text{Experimental yield}} \quad (6)$$

Furthermore, a general (mixed parametric) model expressing the relationship between ester yield and the two independent variables of time and temperature was developed using NLREG version 6.3. software. Several combinations of the independent variables were tested, but the best regression model obtained for this function is as shown in the following equation:

$$EY = -341.5373 + 1.5116\theta - 0.0032\theta^2 - 0.1212T^2 - 0.0180\theta T + 13.9156T \quad (7)$$

where T is the temperature (°C); and θ is time (minutes).

For this model, the coefficient of multiple determination, R^2 has a value of 0.662.

A comparison of the experimental and calculated values of the ester yield generated using Eq. (7) is presented in Table 6. It shows that the deviations are slightly higher than those obtained using the models derived at different temperatures (temperature-specific models). This is expected in view of the lower coefficient of multiple determination of Eq. (7) in comparison with Eqs. (1)–(5).

4.4. Testing of the developed models

In order to properly test the general regression model developed in this work, ester yields at ten new treatment combinations not used in developing the model were calculated and compared to actual experimental yields obtained at those points (Table 7).

T -test was used to compare the two data sets using the procedure given by Obi [21]. The obtained value of 0.04 was lower than the tabulated value of 1.83, which shows that their means were not significantly different at the 5% probability level.

5. Discussion

The results (Table 3 and Fig. 2) show that 60 °C is not a suitable temperature level for base catalyzed neem oil methanolysis at the conditions studied. This is due to the fact that the FAME yield was below 80% irrespective of reaction time. Indeed, apart from 30 min, all other reaction durations resulted in ester yield values below 75%. In the case of 40 °C, it was necessary to run the test for as long as 120 min to achieve 80% conversion. At all other levels of reaction duration lower than 120 min, the corresponding ester yields were lower than 80%. The optimum reaction temperatures for neem oil methanolysis in the presence of 1% NaOH catalyst were 50 °C and 55 °C and for these temperature levels, the optimal reaction

Table 5
Comparison of calculated and experiment values of ester yield.

S. no.	Reaction Temp. (°C)	Duration (min)	Experimental EY (%)	Calculated EY (%)	Deviation (%)
1	40	30.0	45.0	46.3	2.9
2	40	52.5	55.0	53.0	–3.6
3	40	75.0	62.0	60.5	–2.4
4	40	97.5	65.0	69.0	6.2
5	40	120.0	80.0	78.3	–2.1
6	45	30.0	58.0	57.5	–0.9
7	45	52.5	58.0	60.6	4.5
8	45	75.0	68.0	63.4	–6.8
9	45	97.5	63.0	66.4	5.4
10	45	120.0	70.0	69.1	–1.3
11	50	30.0	60.0	60.0	0
12	50	52.5	80.0	79.0	–1.3
13	50	75.0	85.0	88.0	3.5
14	50	97.5	90.0	87.0	–3.3
15	50	120.0	75.0	76.0	1.3
16	55	30.0	62.0	62.6	1.0
17	55	52.5	78.0	76.8	–1.5
18	55	75.0	84.0	84.0	0
19	55	97.5	83.0	84.2	1.5
20	55	120.0	78.0	77.4	–0.8
21	60	30.0	75.0	68.6	–8.5
22	60	52.5	65.0	77.7	19.5
23	60	75.0	70.0	70.4	0.6
24	60	97.5	60.0	46.7	–22.2
25	60	120.0	0.0	6.6	Undefined
Means			66.76	66.76	

Table 6
Comparison of calculated and experimental values of ester yield using Eq. (7).

S. no.	Reaction Temp. (°C)	Duration (min)	Experimental EY (%)	Calculated EY (%)	Deviation (%)
1	40	30.0	45.0	41.9	6.9
2	45	30.0	58.0	57.3	1.2
3	50	30.0	60.0	66.6	-11.0
4	55	30.0	62.0	69.8	-12.6
5	60	30.0	75.0	67.0	10.7
6	40	52.5	55.0	53.8	2.2
7 ^a	45	52.5	58.0	67.1	-15.7
8	50	52.5	80.0	74.4	7.0
9	55	52.5	78.0	75.6	3.1
10	60	52.5	65.0	70.7	-8.8
11	40	75.0	62.0	62.4	-0.7
12	45	75.0	68.0	73.7	-8.4
13	50	75.0	85.0	78.9	7.2
14	55	75.0	84.0	78.1	7.1
15	60	75.0	70.0	71.2	-1.7
16	40	97.5	65.0	67.8	-4.3
17	45	97.5	63.0	77.0	-22.2
18	50	97.5	90.0	80.2	10.9
19	55	97.5	83.0	77.4	6.8
20	60	97.5	60.0	68.5	-14.2
21	40	120.0	80.0	69.9	12.6
22	45	120.0	70.0	77.2	-10.3
23	50	120.0	75.0	78.3	-4.4
24	55	120.0	78.0	73.4	5.9
25 ^a	60	120.0	0.0	62.5	Undefined
Means			66.76	69.63	

^a These values were not used in developing the model.

Table 7
Comparison of calculated and experimental values of test ester yield.

S. no.	Temp (°C)	Time (min)	Exp. EY (%)	Calculated. EY (%)	Deviation (%)
1	40	35	47.0	44.85	4.6
2	42	60	60.1	62.80	-4.5
3	52	80	82.0	79.70	2.8
4	58	72	71.8	74.69	-4.0
5	44	90	68.1	72.78	-6.9
6	60	60	68.2	71.22	-4.4
7	45	50	62.2	66.18	-6.4
8	54	70	82.0	78.36	4.4
9	55	60	79.8	76.76	3.8
10	50	86	84.0	80.00	4.8
Means			70.52	70.73	
Standard deviations			11.92	10.72	

durations were between 60 and 90 min. In all these cases, the obtained ester yields were in the range of 80–90%.

Similarly, reaction durations of 30 min, 60 min and 120 min produced ester yields lower than 80% at all reaction temperature levels studied. The graph (Fig. 2), suggests that reaction durations between 75 min and 100 min, for reactions conducted at temperatures of 50 °C or 55 °C yielded ester yields of nearly 90%.

It is also evident from the graph that there is only a marginal effect in going from 30 min to 52.5 min. The greatest positive effects on ester yield are those between the second and third levels of temperature (45 °C and 50 °C) and time (52.5 and 75 min), respectively. The steep slopes in descent between the fourth and fifth levels of both factors also connote their highest negative effects on the response variable of ester yield. Although, moving from the third to the fourth levels of both temperature (50–55 °C) and time (75–97.5 min) had negative effects on the methyl ester yield, these effects were marginal, judging by the gentle slope between them.

The results are buttressed by practical experience of the transesterification of neem oil, which shows that while lower temperatures or reaction durations are often associated with lower

activation energies; higher temperatures or durations tend to encourage soap formation, thereby reducing the response variable of ester yield in both cases.

Results of the factorial experiments showed that whereas ester yield increased with temperature for the experiments conducted at 30 min, it decreased with increase in temperature for the experiments carried out at 120 min. Furthermore, there was a general increase in ester yield as the reaction duration increased for reactions carried out at lower temperatures of 40 °C and 45 °C but the opposite was the case for reactions conducted at 60 °C. Temperature x time interaction could be responsible for the much higher yields obtained at 50 °C and 55 °C as the reaction time increased from 45 min to 97 min, when compared with 45 °C, for instance. This is also true for the severe negative effects observed on EY at 60 °C with increasing reaction time.

The major contributing factor to the poorer model obtained for the experiments carried out at 60 °C could be the fact that one of the treatment combinations (60 °C for 120 min) yielded insignificant (zero) quantity of biodiesel within a soap mass. In general, 88% of the calculated ester yields were within 7% of the experimental values. This is an indication that NLREG models could be a reliable tool for the estimation of methyl ester yields during base catalyzed transesterification of neem oil.

6. Conclusion

A 5² full factorial experimental design was used to study the influence of reaction temperature and time of neem methyl ester yield in the batch reactor. NLREG Software was used to develop regression models for the ester yield at different temperatures studied. A comparison of ester yields estimated using the models showed promising results. A comparison of the mean ester yield values for ten new treatment combinations estimated using the model with the mean of the experimental ester yield values, showed that they were not significantly different at the 95% level of confidence.

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