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PRODUCTION OF BIODIESEL FROM USED OIL: MODELING, OPTIMIZATION AND RISK ANALYSIS OF THE PROCESS

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ABSTRACT

The competition of biodiesel feedstock with human food chain limits biodiesel production and commercialization; hence, the utilization of waste oil as feedstock holds prospect. This study investigated, modeled, optimized, and accessed the uncertainty and sensitivity of selected process factors (process temperature and time) of biodiesel production from used deep frying oil (UDFO). D-optimal of surface response methodology (RSM) and Monte Carlo simulation (MCS) were used for design and analysis of the UDFO biodiesel production process. Results showed that the average biodiesel yield was 70.13 % v/v. Increment in the process temperature increased the biodiesel yield until an apex after which the biodiesel yield decreased. In addition, increment in process time increased the biodiesel yield throughout the experimental search space. The optimum production condition gave 83.50 % v/v biodiesel yield at process temperature of 63.01^{O}C and process time of 180 min. The validated optimum experimental condition had a percentage error of 0.0743. The certainty of achieving the average experimental biodiesel yield was 87.52%. The biodiesel yield was 87.40 % and 12.60% sensitive to the process temperature and the process time, respectively. It is concluded that biodiesel was successfully produced from UDFO and the established production process mechanism will assist techno-economic development of biodiesel.

Keyword: Biodiesel; Modeling; Optimization; Uncertainty; Risk

Introduction

The world currently has an overwhelming dependence on fossil fuel products for energy generation. This has therefore led to a geometric rise in combustion of fossil fuel products; contributing to increased greenhouse gases and other environmental issues (climate change, global warming phenomenon, ozone layer depletion and extinction of sensitive ecological species, amongst others) that are currently being experienced globally (Nwosu *et al.*, 2017). In addition, the cost of fossil fuel products (petrol, kerosene, diesel and so on) is rapidly increasing worldwide due to exhaustion of the non-renewable natural reserves (Sawyerr *et al.*, 2018), hence, the development and commercialization of green alternative fuel will go a long way to eliminate or reduce the

associated shortcomings of fossil fuel and its products. In contrast to fossil fuel products, the green fuel alternatives are renewable, sustainable and conserve the environment through timely biodegradation when disposed and emission of low or insignificant quantity of environmentally degrading effluents during use (Hoel and Kvemdokk, 1996).

Amongst the currently available green fuels, biodiesel is an important type and it is the alternative vegetative fuel for running diesel engines (Ayouba *et al.,* 2016). It is made from plant oil or animal fat through a process that reduces its viscosity and increases its volatility (Refaat *et al.,* 2008). Having come from natural origin, biodiesel are non-toxic, biodegradable, renewable and sustainable. Technically, biodiesel fuel possesses acceptable qualities or characteristics (for instance, cetane number, viscosity, cloud point and flash point) that are close to synthetic diesel fuel if properly developed (Mata and Martins, 2010). The production methods for biodiesel include blending of oils, micro-emulsion, pyrolsis and trans-esterification (Refaat *et al.,* 2008), however, trans-esterification method is simple to attain. In trans-esterification method, feedstock (fat or oil) is reacted with an alcohol such as methanol or ethanol in the presence of strong homogeneous or heterogeneous catalyst to produce fatty acid methyl esters (biodiesel) and glycerine.

Although biodiesel is the main target of the process, however, the glycerine product is also useful in pharmaceutical industries for the production of cosmetics and soap, and therefore, the process does not give a significant effluent. The stoichiometric reaction of trans-esterification requires one mol of a triglyceride to three mol of the alcohol (Refaat *et al.,* 2008 and Mata and Martins, 2010); however, the usage of excess alcohol is encouraged. When excess alcohol is used, foreword reaction is extended beyond the reverse reaction thereby increasing the yields of the alkyl esters and phase separation of glycerol from biodiesel (Seecharan *et al.,* 2009). A suitable biodiesel for powering engines must have completed its chemical reactions, and be free from glycerine, catalyst, alcohol and free fatty acids (Adepoju *et al.,* 2018).

Although the production and use of biodiesel is desirable, however, the associated high cost of its feedstock and feedstock's direct competition with human food chain is limiting the biodiesel commercial production prospects (Mata and Martins, 2010). Therefore, if the full commercialization potentials of biodiesel are to be realized, the applications of low cost, high oil yielding and non-human food chain competing feedstock alternatives are desirable. Based on the realization of this fact, studies have applied low valued, non-edible or unwanted waste oil including used frying oil, waste-cooking oils and recycled restaurant greases to produce quality and cost effective biodiesel fuel (Ayouba *et al.,* 2016). This feat has led to conversion of waste to wealth and also assisting in reducing environmental pollution from illicit waste oil disposals.

In related studies, Refaat *et al.,* (2008), Seecharana *et al.* (2009), Ayouba *et al.* (2016), amongst others, modeled, predicted and optimized the trans-esterification process of used or waste vegetable oil leading to derivation of quality and low cost biodiesel fuel. However, the evaluations of risk that are associated with the trans-esterification process (output uncertainty and factors

sensitivity analysis) are scarce in the literature and currently remain a gap in study. Therefore, the aim of this study is to fill this lacuna. The specific objectives of the present study are to: (1) design and optimize the trans-esterification process of biodiesel from used deep frying oil (UDFO) using D-optimal response surface methodology (RSM) and (2) investigate the risk (output uncertainty and factors sensitivity) of the trans-esterification process.

Materials and Methods

UDFO that are of groundnut oil origin were sourced from local food vendors in Ogbomoso (Latitude and Longitude 8.1227° N, 4.2436° E), Oyo State, and south western part of Nigeria. The food vendors claimed to have used the waste groundnut oil for deep frying of Akara (bean cake), fish, yam and plantain repeatedly for at least three (3) times. The frying temperature measured when some of the food vendor's deep frying process was on-going ranged from 150 to 200 ^{O}C. Laboratory grade methanol and sodium hydroxide (NaOH) were purchased from Ojota Chemical Market in Lagos State Nigeria. Digital weighing balance (0.00g accuracy) and Stangas dryer were used for measurements and drying purposes, respectively.

Experimental design

Design of experiment is regarded as an important requirement for a scalable and economic process development and RSM is one of its important categories (Singh *et al.*, 2018). RSM is a group of mathematical and statistical methods used for empirical model development, prediction and optimization of the process (Adeyi *et al.*, 2019). In this study, D-optimal type of RSM experimental design useful for quality interpretation of minimized experimental runs was utilized to understand the effect of process variables on the biodiesel yield of UDFO as represented in Table 1.

Table 1 Experimental design for UDFO trans-esterification process

Process Parameter	Unit	Factor Representation	Level		
			Low	Medium	High
Process Temperature	^{O}C	X_1	50	65	80
Process Time	min	X_2	60	120	180
Methanol-Oil Ratio	mol/mol	X_3	1:5	1:5	1:5
Catalyst Loading	g	X_4	0.35	0.35	0.35

The experiment was designed to investigate the effect of process factors and risk of process factors (process temperature and time) on the biodiesel yield of UDFO. Other process variables including methanol–oil ratio (ml) and sodium hydroxide (NaOH) catalyst loading (g) were kept constant. Biodiesel yield was calculated using Eqn. 1 in accordance with the method of Elkady *et al.*, (2015).

$$\text{Biodiesel Yield (\% v/v)} = \frac{\text{Volume of Biodiesel}}{\text{Volume of Trans-esterified Oil}} \text{ X } 100 \quad (1)$$

Purification of UDFO

The cleaning of the used groundnut oil was undertaken before subjecting it to trans-esterification process. The UDFO was increased in temperature to 35 ^{O}C to reduce viscosity and then vacuum filtered with fine cloth sieve (105 µm) to remove solid wastes

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present. Thereafter, the sieved oil was dried in Stangas dryer at 45^{O}C for 12 h to remove associated moisture. The filtered and dried UDFO was subjected to natural cooling in the laboratory environment till it attained room temperature (26 – 28 ^{O}C) before storage in a sealed glass bottle to prevent moisture re-absorption.

Trans-esterification of UDFO

The method of Ojolo *et al.,* (2012) was adopted for the trans-esterification process of UDFO with some modifications. Briefly, a 100 ml of filtered and dried UDFO was taken into a conical flask and preheated to a steady temperature of 40^{O}C through a water bath. A 20 ml of methanol was measured into a beaker and 0.35 g of NaOH was also measured and added to the methanol in the beaker. The contents of the beaker were homogenized at 300 RPM until all the NaOH dissolves completely (about 15 min) in the methanol to form methoxide. The methoxide was then added to the conical flask that contained the preheated UDFO. Thereafter, the temperature of the mixture was adjusted (through the water bath) to the desired process temperature and kept constant for a desired process time in accordance with the experimental design run. After the process time lapsed, the content of the conical flask was further homogenized at 300 RPM for 10 min before it was poured into a separating funnel mounted on a clamp stand and allowed to stand for 24 h. At the expiration of 24 h, biodiesel was separated from glycerin through the funnel outlet. The collected biodiesel was washed twice with equal volume of warm (70 ^{O}C) distilled water to remove water soluble impurities. The recovered biodiesel was then dried in Stangas dryer at 70^{O}C for 5 h, followed by measurement and conversion to percentage biodiesel yield using Eqn. 1. The biodiesel trans-esterification process in this study is as represented in Fig. 1

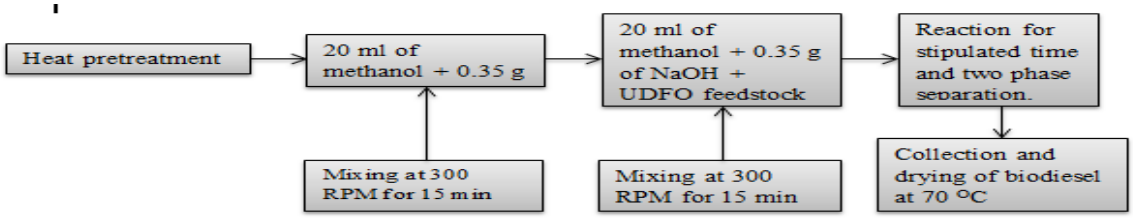


Fig. 1 Trans-esterification process flow in this study

Trans-esterification process modeling

A second order quadratic equation represented in Eqn. 2 was applied to model the process.

$$Y = b + \sum b_i X_i + \sum b_{ii} X_i^2 + \sum b_{ij} X_i X_j \quad (2)$$

Where Y is the estimated parameters response (percentage biodiesel yield); equation coefficients were represented by b (constant term), b_i (linear effect), b_{ii} (quadratic effect) and b_{ij} (interaction effect). The X_i and X_j variables are the trans-esterification process temperature (^{O}C) and process time (min). The model was further analyzed for efficiency using the analysis of variance (ANOVA) and other statistical measures including root mean squared error (RMSE), adjusted coefficient of

determination (Adj. R^2) and coefficient of determination (R^2). The ANOVA was conducted at 5% in accordance with the method of (Singh*et al.,* 2018).

Trans-esterification process optimization

The most effective trans-esterification process factors' variables were investigated using RSM. In the optimization set up, the input trans-esterification process factors were set to be within the range of the experimental data while the percentage biodiesel yield was set to be maximized. All the input trans-esterification process factors were given equal weight to ensure equal importance. The optimum trans-esterification process of UDFO was established after simulation.

The established optimum percentage biodiesel yield was validated through four (4) identical experiments and the average is reported as result because of statistical significance. The percentage error between the RSM's derived optimum and experimentally validated optimum transesterification process factors' variables were expressed using Eqn. 3

$$ɖ = \frac{Ve-Vp}{Vp} \text{ X } 100\% \qquad (3)$$

Where $ɖ$ is the percentage error

Ve is the experimental optimum

Vp is the RSM predicted optimum

Risk analysis of UDFO biodiesel production

The uncertainty and sensitivity of trans-esterification process factors on the percentage biodiesel yield was aimed for quantification. Sensitivity analysis was done using the RSM's established mathematical model. To do this, Monte Carlo simulation in Microsoft Excel 2015 Package was employed. Monte Carlo functions through the power of random numbers generation within the defined range of independent variables to construct a probability distribution curve from where the probabilistic analyses are established. Prior to the simulation, the inputs trans-esterification process factors were declared as the assumptions in their respective experimental range of values while percentage biodiesel yield was the forecast variable. Thirty thousand (30,000) iterations were performed to achieve minimum mean standard error in the result. Table 2 shows the selected type of input assumption and range for each trans-esterification process factors that was employed for the simulation in this study.

Table 2 Parameters of risk analysis

Input Factors	Distributions	Range of variables
Temperature (^{O}C)	Uniform	5 – 10
Time (min)	Uniform	12 – 72

Results and Discussion

Effect of trans-esterification process factors

The result of the trans-esterification process is represented in Table 3. The table showed that the minimum percentage biodiesel yield of 62.01 % v/v was achieved at trans-esterification temperature of 80.00 ^{O}C and trans-esterification time of 60.00 h while the minimum percentage biodiesel yield of 83.53 % v/v was achieved at trans-esterification

temperature of 65.00 and trans-esterification time of 180.00 h. The average percentage biodiesel yield stands at 70.13 % v/v. This average percentage biodiesel yield justified the choice of the range of the experimental design and it showed that this range is truly effective for high percentage biodiesel production yield.

Table 3 Experimental percentage biodiesel yield

S/N	Process Temperature (^{O}C)	Process Time (h)	Biodiesel Yield (% v/v)
1	80.00	180.00	62.09
2	65.00	120.00	82.56
3	50.00	120.00	69.72
4	50.00	60.00	70.17
5	50.00	180.00	72.01
6	65.00	60.00	80.54
7	80.00	120.00	62.14
8	65.00	180.00	83.53
9	80.00	60.00	62.01
10	80.00	60.00	62.87
11	50.00	60.00	69.98
12	80.00	180.00	63.89
Average Biodiesel Yield =			70.13

The individual and interaction effect of the trans-esterification process factors on the percentage biodiesel yield is represented in Fig. 2.

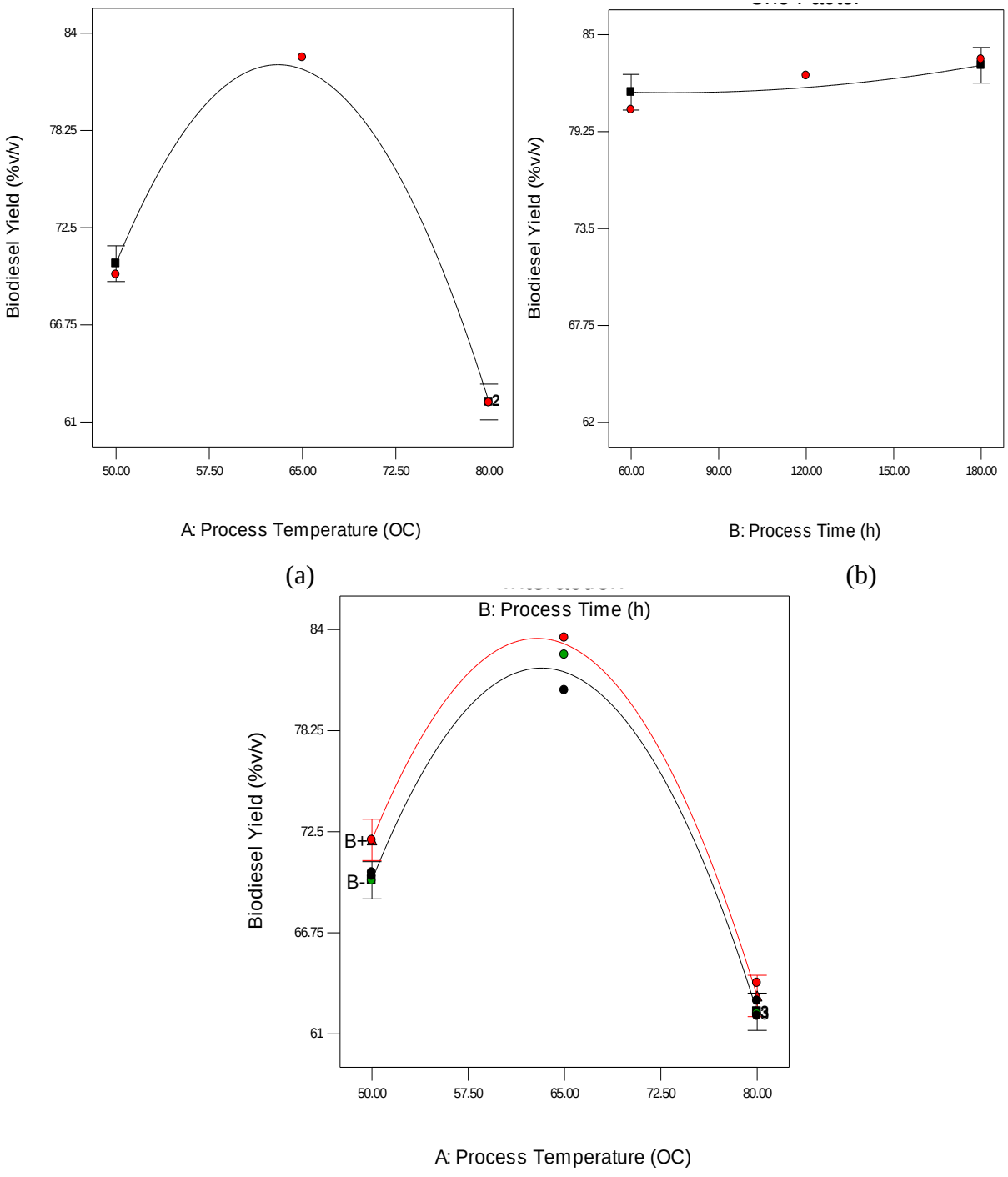


Fig. 2 Effect of trans-esterification (a) process temperature, (b) process time and (b) time - temperature interaction of the percentage biodiesel yield of UDFO

Fig. 2 (a) showed that trans-esterification temperature had a concave dependence on the percentage biodiesel yield with minimum, maximum and peak values at 69.02, 81.79 and 0.00 % v/v, respectively. The observed downward trend after the peak is attributed to degradation of reaction constituents at extreme temperatures. A close report was also made by Ogunkunle *et al.,* (2017) when biodiesel was produced from Milk Bush Seed (*Thevetia peruviana*) Oil using both heterogeneous and homogeneous catalyst. Fig. 2 (b) showed that the trans-esterification time had an increasingly positive effect on the percentage biodiesel yield throughout the experimental search points. This means that the more the trans-esterification reaction or process time the better the percentage biodiesel yield becomes. This can be attributed to strong initial bond between the biodiesel and glycerin components of UDFO, and as such, longer reaction times are needed to disintegrate the bond and split UDFO into biodiesel and glycerin components. This result conforms to the report of Adepoju *et al.,* (2018) on the investigation of Brette Pearl Spar Mable (BPSM) as a renewable catalyst for biodiesel production from *Thevetia peruviana* seed oil. Fig 2 (c) showed the interaction between the trans-esterification process factors. The figure showed that there was no interaction (crossing of curves or trends) between the two trans-esterification process factors. The tendency of interaction almost occurred towards the end of the trans-esterification process; however, this did not occur. This showed that the process factors independently affected the trans-esterification process and not interactively. In the entire figure, the oval shaped bullet represents the experimental values while the rectangular shaped bullet represents the predicted values. The beam shaped bullet represents the least square difference (LSD). The close arrangements of the bullets types in the figure 2 (a), (b) and (c) a showed that both the experimental and predicted values are close showing the efficiency of the RSM developed model.

Modeling and analysis of variance of the trans-esterification Process

The statistical model developed for the biodisel prodcution process in this study using RSM is represented in Eqn. 4

$$BY = -195.77657 + 8.78610 * A + 2.69354E\text{-}003 * B - 0.069317 * A^2 + 1.52408E\text{-}004 * B^2 - 4.01143E\text{-}004 * A * B \quad (4)$$

Where BY = Biodiesel Yield
A = Process Temperature
B = Process Time

A glance at the model showed that the process temperature (second term of the equation) had a positive contribution to the process while the square of process temperature (fourth term of the equation) had a negative contribution to the process. This is in harmony with the result observed in Fig. 2 (a) where low process temperature improved percentage biodiesel yield while high process time above a peak reduces the percentage biodiesel production. Furthermore, both process time (third term of the equation) and square of process time (fifth term of the equation) had a positive contribution to percentage biodiesel production.

This result is also in harmony with the result observed in Fig. 2 (b) where the process time continually contributes to the percentage biodiesel yield at all investigated process times. The model interaction term (sixth term of the model) showed a negative contribution

to the percentage biodiesel yield. This is confirms the fact that no interaction of trans-esterification process occurred as established in Fig. 2 (c). The analysis of variance of the model is represented in Table 4.

Table 4 Analysis of variance (ANOVA) for the trans-esterification process

Source	Sum of Squares	DF	Mean Square	F-Value	Prob > F	
Model	728.65	5	145.73	193.16	< 0.0001	significant
A	142.45	1	142.45	188.80	<0.0001	
B	5.32	1	5.32	7.05	0.0378	
A^2	534.44	1	534.44	708.36	<0.0001	
B^2	0.66	1	0.66	0.88	0.3853	
AB	0.85	1	0.85	1.13	0.3289	
Residual	4.53	6	0.75			
Lack of Fit	2.51	3	0.84	1.24	0.4318	not significant
Pure Error	2.02	3	0.67			
Cor Total	733.18	11				

The Model F-Value of 193.16 from Table 4 implies that the model is significant. There is only a 0.01% 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, B and A^2 are significant model terms. Values greater than 0.1000 indicate the model terms are not significant. The 'Lack of Fit F-value' of 1.24 implies that the Lack of Fit is not significant relative to the pure error. There is a 43.18% chance that a 'Lack of Fit F-value' this large could occur due to noise. Non-significant lack of fit is good because we want the model to fit. Other performance metrics of the model is represented in Table 5.

Table 5 Statistical performance metrics of the fitted model

Performance Metrics	Value
R-Squared	0.9938
Adj R-Squared	0.9907
Pred R-Squared	0.9741
Adeq Precision	38.867

The R-Squared of 0.9938 is close to unity (1) and therefore satisfactory (Adeyi, et al., 2021). The 'Pred R-Squared' of 0.9741 is in reasonable agreement with the 'Adj R-Squared' of 0.9907. 'Adeq Precision' measures the signal to noise ratio. A ratio greater than 4 is desirable. The value of 38.867 indicates an adequate model signal. This model can be used to navigate the design space satisfatorily.

Optimization of trans-esterification process

The input process factors variable that will likely result in maximum percentage biodiesel in this study is represented in Fig. 3. The Fig. showed that a process temperature 63.01^{O}C

and a process time of 180.00 min will give a percentage biodiesel yield of 83.50 % v/v with a desirability (accuracy) value of 0.999 which is close to the desired unity (1) value (Oke *et al.*, 2017).

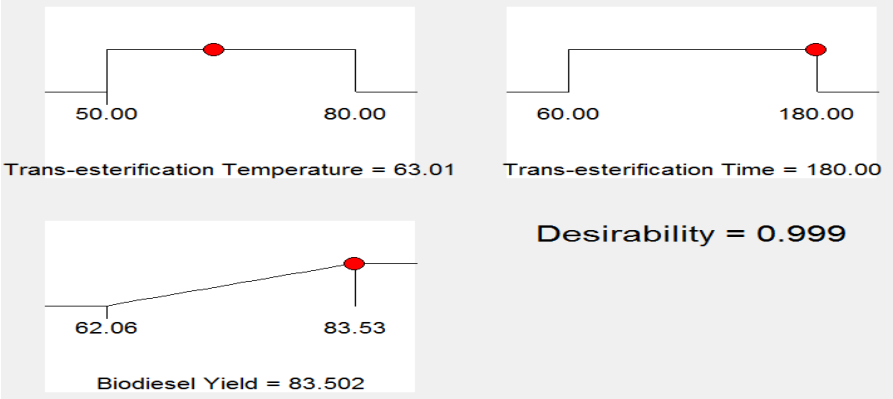
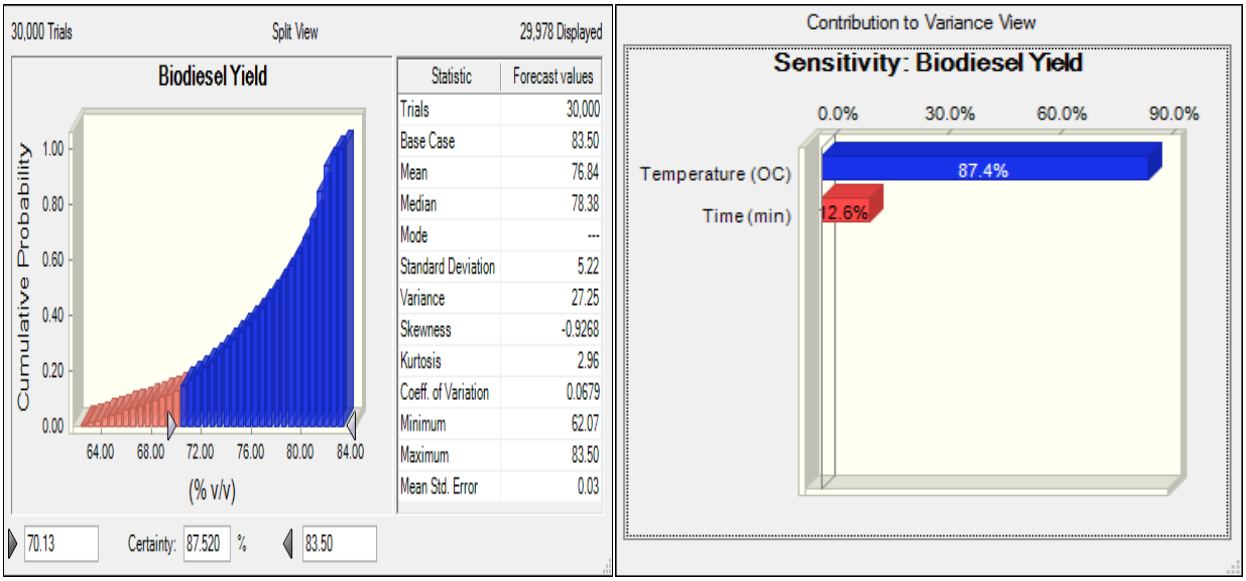


Fig. 3 Optimum percentage biodiesel yield

The mean of the four (4) identical experiment conducted to validate this RSM's established theoretical optimum percentage biodiesel production condition gave 83.44 % v/v. Comparing the validated and theoretical percentages of biodiesel yield in this study, it is established that a percentage error of 0.0743 occurred within the values. This error is considered insignificant and may be associated with uncontrolled laboratory conditions.

Risk analysis of the trans-esterification process

The certainty of achieving the experimental result is represented in Fig. 4 (a). The figure displays the result of 29,987 solutions out of the 30,000 trials being the default display range of OCB that includes all trials within 2.6 standard deviations of the mean (approximately 99% of the forecast values).



(a) (b)

Fig. 4. (a) Uncertainty analysis and (b) sensitivity analysis of the percentage biodisel yield

The result was derived using the RSM's suggested optimum condition as the base case for the Monte Carlo simulation. This shown in the statistics section of the figure. The risk of achieving at least the average percentage biodiesel yield of 70.13 % v/v that was established in Table 3 gave a certainty of 87.52% as shown in the buttom of the figure. Technically, this value is high and it gives credence to the reliability of the experimental data.

Furthermore, the associated risk with each of the trans esterification process factors is shown in Fig. 4 (b). The figure displayed the contribution to variances by the process factors during the biodiesl production.The figure showed that process temperature had the most contribution to changes that accompanies the process with a sensitivity value of 87.40% while the process time contributes the least to the variances of the process with a sensitivity value of 12.60%. It therefore means that process temperature is the greatest risk here and its handling may make or mar the percentage biodiesel yield.

Conclusion

This study investigated, modeled, and optimized the biodiesel production from UDFO. In addition, the study accessed the associated risk of the process factors that are involved in the trans-esterification production of biodiesel from UDFO for increased biodiesel production benefits. The methodologies applied are design of experiment and Monte Carlo Simulation. Findings showed that the average biodiesel production yield was 70.13 % v/v. Increment in process temperature positively increased the percentage of UDFO biodiesel yield until an apex after which increment in process temperature decreased the percentage biodiesel yield. On the other hand, increment in process time had an interrupted increase in the percentage biodiesel yield throughout the experimental search space. The optimum

trans-esterification condition for biodiesel yield was 63.01 process temperature and 180 process time to give 83.50 % v/v percentage biodiesel yield. The validation experiments for the predicted optimum trans-esterification condition had a percentage error of 0.0743. The certainty of achieving the average experimental percentage biodiesel yield was 87.52%. The percentage biodiesel yield was 87.40% sensitive to trans-esterification process temperature and 12.60% sensitive to process time, respectively. It is concluded that biodiesel was successfully produced from UDFO. The model developed, optimum process factors, uncertainty and sensitivity values determined are important for trans-esterification process mechanism understanding, process design and control and product standardization. It is recommended that the optimum and sensitivity values here derived, be applied for further techno-economic study of the UDFO trans-esterification process for commercialization.

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