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Brette Pearl Spar Mable (BPSM): a potential recoverable catalyst as a renewable source of biodiesel from *Thevetia peruviana* seed oil for the benefit of sustainable development in West Africa

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Abstract

Background: The energy requirements are globally on a rapid escalation, as technology advances, which is also true for a developing country like Nigeria, which is dependent on fossil fuels and its derivatives. Apart from its adverse effect on its economy, it has also negative impacts on the health and the environment, in general. However, investments in renewable energy are faced by the competitive oil prices, the very high investment cost for renewable energy, and high local electricity prices. This paper appraises the attractiveness of investing in renewable energy sources over the continued use of non-edible oil for electricity generation.

Methods: This paper explores the application of biomass seed oil to produce a renewable fuel (biodiesel) using heterogeneous base catalyst. Meanwhile, two-step processes were employed to produce the biofuel. In the first step (esterification), the acid value of the oil was reduced to the recommended limit (FFA ≤ 1.5) using H_2SO_4, while in the second step (transesterification), the catalyst calcination of grounded Brette Pearl Spar Mable (BPSM) pre-soaked in methanol was used as a biobase for biodiesel production. For the optimization, minitab response surface (MRS) and artificial neural network (ANN) were employed to model and optimize the process variables responsible for the optimum production of the oil and the biodiesel.

Results: The result presented showed that *T. peruviana* seed was found to be rich in oil with an average yield of 44.00% (*w/w*), and the oil was highly unsaturated with a high FFA. The maximum experimental biodiesel yield obtained was 86.00% at a catalyst amount of 4 g, a reaction time of 70 min, and a methanol/oil ratio of 0.1(*v/v*). This result was validated in triplicate under the same conditions, which yielded 85.70% (*v/v*) for MRS and 85.98% (*v/v*) for ANN. Furthermore, the optimization results also indicated that the p values ($p < 0.05$) of the model terms were significant, and the accuracy of the models achieved by MRS and ANN based on R^2 depict that both optimization tools gave good predictions of R^2 (MRS: $R^2 = 99.98\%$ and ANN: $R^2 = 99.97\%$). The properties of the biodiesel, as described in other earlier reports using the same feedstock with different catalysts, indicated that the produced biodiesel had properties which agreed to those reported in the literature.

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Conclusions: *T. peruviana* seed has proved to be a good biomass raw material for oil production, and its conversion to biofuel using a heterogeneous biobase catalyst showed its suitability as a renewable environmental friendly fuel. Government should invest in more sustainable sources of energy by imposing law for the use of non-edible oil or decreasing the price of electricity.

Keywords: *T. peruviana* oilseeds, Minitab response surface (MRS), Artificial neural network (ANN), Brette Pearl Spar Mable, Catalyst calcination, Esterification, Transesterification

Background

In recent decades, biodiesel has blossomed as a greener and new renewable alternative to petroleum diesel, which is a non-toxic, non-aligned, carbon, sulfur, and aromatics free fuel [1, 2]. Biodiesel can be said to be an option to conventional diesel which is transformed from vegetable oils in the presence of a primary alcohol and homogeneous catalyst, typically followed by base transesterification of naturally occurring triglycerides [3]. It has been reported that biodiesel and petroleum diesel have similar physicochemical properties in terms of their performance in an engine as a fuel [4]. Furthermore, it has also been established that, biodiesel blended along with up to 20% conventional diesel, could be used as a fuel for diesel engines [5].

Prior to the transesterification, oils were usually extracted from oil seeds through various methods such as mechanical [6–8], pressurized solvent extraction [9], soxhlet extraction [10], ultrasonic extraction [11, 12], aqueous enzymatic oil extraction (AEOE) [13, 14], stirring and shaking extraction [15] to mention a few. These methods have been reported to be used for the extraction of vegetable oils and other plant essential components, each with intrinsic advantages and shortcomings [16]. Mechanical pressing is not only the oldest and easiest method widely used, but also a method which produces oil of low value. Meanwhile, in the literature [17], the use of supercritical CO_2 extraction was reported, the oil yield obtained was higher compared to that obtained from using solvent extraction. Though the produced oil has high purity, it also has high operating and investment costs. Solvent extraction, however, has various advantages, including high yield, less turbidity, as well as environmentally friendly and cost-effective properties [18, 19] as in the case of an extraction of oil from *Jatropha curcus*, described by Kim et al. [20]. All three, Umer et al. [21], Betiku et al. [22], and Adepoju et al. [23], studied the solvent extraction of oil from *Moringa oleifera* and sorrel seed and *Chrysophyllum albidium* oilseeds, respectively. In addition, Adepoju et al. [23] also examined the quality characterization of *Moringa oleifera* oil. In another study, Betiku et al. [24] investigated the solvent extraction of oil from Beniseed (*Sesamum indicum*) oilseeds. These oils however have already been applied as feedstocks in many applications, such as paint, pharmaceuticals, cosmetics, and others.

The use of seed oil has already been demonstrated by Rudolf Diesel, but owing to its high viscosity, partial ignition, deposition of particulate matters and lower volatility, it cannot suitably work like a conventional fuel in a modern engine designed for modern fuel, [25]. Hence, different techniques such as pyrolysis [26], microemulsion [27], and transesterification [28] have been applied to overcome these problems. Among these, transesterification has been studied and reported to be most effective, time-saving and economical, when blended or used directly in either a modern engine or diesel fuel engine, respectively [29, 30].

Nevertheless, transesterification of biodiesel produced from the refined edible oil is not viable, owing to food threats and high production cost. Therefore, non-edible vegetable oil is a good alternative to overcome the problems with regard to food threat and the lowering of production cost [2]. The use of non-edible oilseeds such as *Azadirachta indica*, *Jatropha curcas*, *Jatropha gossipifolia*, *Calophhyllum inophyllum*, *Hura crepian*, *Chrystophyllum albidium*, and *Thevetia peruviana* have been extensively discussed in the literature [31–35]. Although some of these oils are characterized by high amounts of free fatty acid (FFA), they are an alternative for the cost reduction of the transesterification process. Oils characterized by high amounts of FFA can be esterified using an acid of a lower pH value (≤ 1.5) before their transesterification by alkaline/heterogeneous base catalysts. When choosing calcinated potassium silicate oils characterized by high amounts of FFA, they can easily be converted without esterifying, but due to the high cost of potassium silicate, the first solution is better than the latter.

Homogeneous and heterogeneous catalysts have reportedly been applied to biodiesel production. Dhoot et al. produced biodiesel using a homogeneous alkali catalyst, whereas Ana et al. [36] have used a heterogeneous mixture of Hexane/Ether as a catalyst for producing biodiesel and Betiku et al. [37] a homogeneous catalyst for producing biodiesel from *Sesamum indicum* oil. A methanolysis of soybean oil with 1% potassium hydroxide as a catalyst has been carried out by Tomasevic et al., whereas Umer et al. have worked on the application of a response surface methodology for the optimization of the transesterification of *Moringa oleifera* oil applying a homogeneous catalyst, and Zhang et al. [38] have worked on the

optimization of the transesterification reaction from cotton seed oil by means of a statistical approach using a homogeneous base catalyst. The major drawbacks of these types of catalysts are difficulties in their recovery from the reaction medium and attendant problems, in particular saponification, excess reactant consumption, environmental pollution, high oil/alcohol molar ratios, and extra costs for separation, which increase the overall costs of production [35]. Heterogeneous catalysts mainly involve the use of a solid base catalyst, which can easily be separated from the transesterified product, and is always active at the boiling point temperature of methanol during transesterification [39]. In support of this claim, Choi [40] reported that the heterogeneous catalyst is recoverable, less corrosive, produces no soap, and can be reused. Therefore, researchers have tested varieties of solid base for the catalytic activity. For this purpose, Xie et al. [41] prepared potassium loaded on alumina as a solid base catalyst and employed it for the transesterification of soybean oil at reflux of methanol. Under the same conditions, they examined the solid base catalysis of magnesium-aluminum (Mg-Al) mixed oxide prepared by calcining the corresponding hydrotalcite. Interestingly, Shibasaki-Kitagawa et al. [42] have explained the anion-exchange resin catalyzed through a transesterification of triolein, which is a model of vegetable oil. In the same vein, Deka [43] have used *M. balbisiana* as a heterogeneous catalyst for the production of biodiesel from yellow oleander. Several studies described in [35, 44, 45] have also examined the use of different heterogeneous catalysts in the production of biodiesel with or without optimizing the process conditions.

Minitab is a statistical analysis software package that provides a wide range of data analysis options and has been used in optimization conversion of sunflower oil to biodiesel using sodium methoxide. Data analysis was performed through response surface methodology (RSM) and by using Minitab v.14, statistical package [45]. Yan [46] developed linear and full quadratic regression models using Minitab version 16 software, both to predict FFA and FAME concentration and to optimize the reaction conditions of biodiesel production from *Calophyllum inophyllum* oil. In the same vein, biodiesel production from *Scenedesmus sp*. through optimized in situ acid in a transesterification process has been carried out by Xie [40]. The study used Minitab RSM version 16 to examine the process analysis. The advantages of this software over others include accessibility, strength in statistical quality control, descriptive and inferential statistics, statistical process control (SPC), reliability, gage repeatability and reproducibility (GR&R) studies, process capability, and better graphing output [47].

Artificial neural network (ANN) is a learning system based on a computational technique, which shows a non-linear relationship between connecting factors and actual responses by means of iterative training of data obtained from a designed experiment [48]. ANNs show superiority as a modeling technique for data sets, data fitting, and prediction abilities [48, 49], which has also been applied in various professional fields of engineering, health, and sciences [50]. An early report presented in [51] ANN was applied to determine diesel engine performance and exhaust emission analysis using waste cooking biodiesel fuel. The use of ANN for the prediction of the cetane number of biodiesel has been presented in [52]. Furthermore, a based prediction of performance and emission characteristics of a variable compression ratio CI engine, using WCO as a biodiesel at different injection times by means of ANN was examined by Shivakumara et al. [53], whereas Najafi et al. [54] have applied ANN in the performance and exhaust emissions of a biodiesel engine, and Najafi [55] carried out a combustion analysis of a CI Engine Performance employing waste cooking biodiesel fuel with an ANN Aid. Another application includes not only biodiesel production from soybean oil [56] and waste frying palm oil [57], but also the prediction of engine performance for an alternative fuel [58] and the simulation of biodiesel production from waste olive oil [59].

Therefore, the aim of this study is to exploit the use of calcinated BPSM for biodiesel production from *Thevetia peruviana* (*T. peruviana*) in a two-step conversion. To determine the significance of the variables for the processes, minitab response surface and artificial neural network will be used for modeling and optimizing the process variables. The fuel properties of *T. peruviana* biodiesel will also be evaluated with the objective of determining its potential use.

Methods

Materials

Method for the extraction of T. peruviana powder oil

Matured oilseeds of *T. peruviana* were collected from the Ogbo Grammar School, Omu-Aran, Kwara State, Nigeria. The greenish fleshy pericarps were removed manually, while the hard brownish mesocarp with the seeds was sun-dried to constant weight. The kernel fruit was shelled and sun-dried until constant weight. Separation of chaff from the kernel fruit was carried out by winnowing. The cleaned kernel fruit was then milled into powder (*T. peruviana* oilseed powder) and kept in a clean container for further processing.

The BPSM used was obtained from the Landmark University, Omu-Aran, Kwara State, Nigeria. The BPSM was not only washed in distilled water to remove dirt, but also sun-dried to constant weight before grounding to a particle size of 5 mm (to ensure a large surface area per unit mass), and stored in a clean crucible for further processing. The necessary chemicals hexane, ethanol, Wij's solution, diethyl ether, chloroform, and others were purchased from Isolak Nig. Ltd. and were of "organic trace analysis" grade.

Methods

Method for the extraction of T. peruviana powder oil

A known weight of *T. peruviana* oilseed powder was placed in a muslin cloth, and analytical grade n-hexane was employed as a solvent for extraction. According to optimization software, 15 experimental runs were generated and carried out in a 4-faced heating mantle capable of holding 500 ml capacity Soxhlet extractors. Excess n-hexane in the oil was recovered using a "rotavaps" (rotary evaporator), and the percentage yield of oil produced was calculated based on the ratio of weight oil extracted to that of the weight of *T. peruviana* powder used (Eq. 1).

$$T.peruviana\ oil\ yield\ (\%) = \frac{weight\ of\ T.peruviana\ seedoil}{weight\ T.peruviana\ oilseed\ powder} \times 100 \quad (1)$$

Experimental design of oil extraction from T. peruviana oilseed powder

In order to model and optimize the extraction of oil from *T. peruviana* powder, minitab response surface (MRS) version 15.5 was employed to generate 15 experimental runs. The influence of extraction time: X_1, solvent volume: X_2 and sample weight: X_3 were considered (Table 1). The experimental runs were randomized to minimize the effects of unexpected variability in the observed responses, and the process was described using a second order model. To relate the response variable to the independent variables, multiple regressions were introduced to fit the coefficient of the second-order quadratic polynomial model of the response. The fit of the model was evaluated using a test of significance and ANOVA analysis.

The properties of extracted T. peruviana seed oil

The properties of the extracted oil were evaluated based on physical, chemical, and other properties employing standard methods (AOAC 1997) and Wij's method.

Gas chromatographic mass spectrometric (GC-MS) analysis of T. peruviana seed oil

To ascertain the compositions of free fatty acid present in the oil, a gas chromatographic mass spectrometer (Agilent 19091S-433HP-5MS) was employed. The operating conditions of the equipment were as follows: Column Elite-1 fused silica capillary column (30 mm × 250 μm × 0.25 μm, composed of 5% phenyl methyl silox), operating in an electron multiplier volts 1329.412 eV, Helium (99.99%) was applied as a carrier gas at a constant flow of 1.5 ml/min, and an injection volume of 1 μl was employed (split ratio of 10:1), at an injector temperature of 150 °C and an Ion-source temperature of 250 °C. The oven temperature was programmed from 35 °C (isothermal for 5 min.), at an increase of 4 °C/min to 150 °C, for 2 min, then at 20 °C/min to 250 °C, for 5 min (isothermal at 250 °C). The mass spectra were taken at an average velocity of 44.297 cm/s at a hold up time of 1.1287 min, a pressure of 11.604 psia and a frequency of 50 Hz with a total GC running time of 45 min.

Catalyst calcination and elemental characterization

Eight samples, each weighing 50 g of grounded BPSM were prepared. Four samples were soaked in methanol for 10 min and were stirred before filtering, while the remaining four samples were not soaked. The eight samples were calcinated in a Carbolite AAF 1100 furnace at 700 °C for 4, 5, 6, and 7 h, respectively. To identify the calcinated BPSM with the highest base catalyst, elemental analysis of the calcinated samples was performed using an EDXRF Spectrophotometer (EDX3600B). The EDXRF spectrophotometer was calibrated according to the recommended silver standard, while the analysis was carried out using the ore standard calibration curve. The calcinated BPSM identified with the highest base catalyst was used for the second-stage transesterification process.

The production of seed oil biodiesel by T. peruviana

The initial acid value of *T. peruviana* seed oil amounted to 3.8048, corresponding to an FFA level of 1.9024, which was above the limit for a satisfactory transesterification. Hence, a reduction in the acid value via an acid-catalyzed esterification process was required.

Acid-catalyzed esterification process

An acid-catalyzed esterification process was carried out according to the modified method adopted by [35]. 75 ml of *T. peruviana* seed oil were measured and then were filled into a 250 ml glass reactor for

Table 1 Variables factors considered for *T. peruviana* oil extraction

Variable	Symbol	Coded factor levels		
		-1	0	+1
Extraction time (min)	X_1	40	50	60
Solvent volume (ml)	X_2	200	225	250
Sample weight (g)	X_3	40	45	50

heating to 60 °C on a hot plate using a magnetic stirrer for preheating, and mixing a known volume of acid (H_2SO_4) with a known volume of an analytical grade methanol. After this, the mixture was added to the pre-heated oil in the reactor and was allowed to stir continuously for 1 h. The esterified oil was then transferred into a separating funnel to stand for 2 h. The methanol-water layer formed on the bottom of the separating funnel was tapped. Excess methanol in the oil was evaporated prior to the determination of the acid value. This process was repeated regularly under different conditions until the minimum acid value reached 0.7896 mg KOH g^{-1}, which corresponded to FFA = 0.3948 mg KOH g^{-1} before the transesterification process.

Base-catalyzed transesterification of esterified oil

The base-catalyzed transesterification of esterified oil was carried out according to the modified method of [60]. Hence, the transesterification procedure of *T. peruviana* seed oil was performed, as requested. A known volume of esterified *T. peruviana* seed oil with the lowest acid value was measured and introduced into a 250-ml-necked reactor with a magnetic stirrer, where it was placed on a hot plate, and charged with a known weight of base catalyst dissolved in a known volume of anhydrous methanol. Two layers were observed, and the reactor flask was covered by the stopper to prevent the methanol from escaping, as the reaction proceeded. The hot plate temperature regulator was then adjusted to 60 °C to complete the reaction at a known time. At the end of the reaction, the resulting products were transferred to a separating funnel for glycerol (denser) and biodiesel (less dense) gravity settling (24 h). Glycerol was tapped off from the bottom of the separating funnel leaving behind the less dense biodiesel in the funnel. Meanwhile, the clarity of the biodiesel has significant effects on its fuel properties. The biodiesel left in the funnel was then washed until the distilled water became clear, in such a way allowing the residual catalyst, untapped glycerol, methanol, and soap to be removed. The washed *T. peruviana* biodiesel was then tapped into a pyrex flask, where it was further dried over heated $CaCl_3$. After this, the dried biodiesel was purified by filtration, and the final *T. peruviana* biodiesel was calculated in terms of % (*v*/*v*), as presented in Eq. (2). This experiment was repeated for 15 runs as had been modeled by design software.

$$T.peruviana\ biodiesel\ yield\%(v/v) = \frac{Volume\ of\ T.peruviana\ \text{biodiesel}}{Volume\ of\ esterified\ T.peruviana\ oil} \quad (2)$$

Experimental design for the transesterification of esterified *T. peruviana* oil

To optimize the transesterification of esterified *T. peruviana* oil, the minitab response surface (MRS) version 15.5 was used to generate 15 experimental runs. The factors selected for the transesterification process were methanol/oil ratio: X_1, reaction time (min): X_2, and catalyst amount (g): X_3 (Table 2). The experimental runs were randomized to minimize the effects of unexpected variability in the observed responses. The process was described by the second order method employed. To relate the response variable to the independent variables, multiple regressions were used to fit the coefficient of the second-order quadratic polynomial model of the response. The quality of the fit of the model was evaluated using both a test of significance and ANOVA analysis.

Properties of *T. peruviana* biodiesel

It is necessary to determine the properties of the biodiesel produced from the *T. peruviana* seed oil to ascertain its suitability for running an internal combustion engine (IC engine). These properties include, among others, the specific gravity, kinematic viscosity, moisture content, peroxide value (PV), saponification value (SV), and iodine value (IV).

Fuel properties of biodiesel

The fuel properties of the produced biodiesel, such as cetane number (CN), higher heating value (HHV), API value, diesel index, and aniline point, were computed applying the standard Eqs. (3–7) according to American Society of Testing and Material (ASTM) D2015.

Table 2 Variables factors considered for transesterification of *T. peruviana* oil to biodiesel

Variable	Symbol	Coded factor levels		
		-1	0	+1
Catalyst amount (g)	X1	2.00	4.00	6.00
Reaction time (min)	X2	50	60	70
Methanol/oil ratio (v/v)	X3	0.10	0.15	0.20

$$Cetane\ no. = 46.3 + \frac{5458}{Saponification\ value} - 0.225\ Iodine\ value \quad (3)$$

$$HHV\ (MJ/kg) = 49.43 - [0.041(Saponification\ value) + 0.015(Iodine\ value)] \quad (4)$$

$$API = \frac{141.5}{Specific\ gravity@15^{o}c} - 131.5 \quad (5)$$

$$Diesel\ index = \frac{Cetane\ number - 10}{0.72} \quad (6)$$

$$Aniline\ point = \frac{Diesel\ index \times 100}{API} \quad (7)$$

Statistical data analysis

The data obtained from the *T. peruviana* oilseed extraction experiment and the transesterification of biodiesel were analyzed statistically using MRS version 15.5 and ANN, a Neural Power version 2.5 (CPC-X Software), in order to fit the quadratic polynomial equation. To correlate the response variable to the independent variables, multiple regressions were used to accommodate the coefficient of the polynomial model of the response. The quality of the fit to the model was evaluated using TS (test of significance) and ANOVA (analysis of variance). A statistical analysis was performed by evaluating the regression model called determination coefficient (R^2), root means square error (RSME) and plotted the 2D contour line as well as the 3D graph. The fitted quadratic response model equation is described in Eq. (8).

$$R_F = \tau_0 + \sum_{i=1}^{k} \tau_i X_i + \sum_{i=1}^{k} \tau_{ii} X_i^2 + \sum_{i<j}^{k} \tau_{ij} X_i X_j + e \quad (8)$$

where R_F denotes the response factor (*T. peruviana* seed oil/biodiesel).

τ_0 represents the intercept value, τ_i ($i = 1,2,k$) the first order model coefficient, τ_{ij} the interaction effect, τ_{ii} the quadratic coefficients of X_i, and e is the random error.

Results and discussion

Optimization of the extraction of *T. peruviana* seed oil

The coded factors considered in this study are listed in Table 3 along with the experimental results, the predicted values, and the residual values obtained. Observations from Table 3 indicate that the highest experimental *T. peruviana* seed oil yield was 44.00% (*w/w*) at variable factors of X_1 = 60 min, X_2 = 225 ml, and X_3 = 40 g. The predicted responses for MRS and ANN were 44.04% (*w/w*) at variable factors of X_1 = 59.30 min, X_2 = 225 ml, and X_3 = 39 g and 43.95% (*w/w*) at variable factors of X_1 = 59.00 min, X_2 = 220 ml, and X_3 = 38.70 g, respectively. The predicted values were validated by carrying out three experiments under the predicted variable conditions to obtain an average *T. peruviana* seed oil yield of 43.50% (*w/w*) and 43.80% (*w/w*) for MRS and ANN, respectively. Table 4 illustrates the results of the test of significance for each regression coefficient. The results showed that the p values (X_1, X_3), (X_1X_3), and ($X_1^2$and X_3^2) of the model terms were significant ($p < 0.05$). The model coefficients and probability values are shown in Table 5, whereas the analysis of variance of the regression equation model is shown in Table 6. The mathematical expression of the relationship between *T. peruviana* seed oil and the variables considered is given in Eq. (9).

$$Y_{Oil}\%(w/w) = -145.0 - 0.027X_1 + 7.02X_3 + 0.0558X_1^2 - 0.01692X_3^2 - 0.110X_1X_3 \quad (9)$$

Table 3 Coded factors with experimental oil results, predicted and residual values by MRS and ANN for *T. peruviana* oilseed extraction

Std run	X_1	X_2	X_3	Oil yield% (v/v)	Predicted		Residual	
					MRS	ANN	MRS	ANN
1	0	0	0	28	27.38	27.33	0.62	0.67
2	0	0	0	27	27.38	27.33	-0.38	0.33
3	0	1	1	27	26.96	27.01	0.04	0.01
4	0	-1	1	27	26.96	27.01	0.04	0.01
5	1	0	1	33	33.04	32.97	-0.04	0.03
6	-1	0	1	32	32.04	32.00	-0.04	0.00
7	0	0	0	27	27.38	27.33	-0.38	0.33
8	-1	-1	0	27	26.96	27.00	0.04	0.00
9	1	1	0	39	38.96	39.02	0.04	0.02
10	1	-1	0	39	38.96	39.02	0.04	0.02
11	1	0	-1	44	44.04	43.95	-0.04	0.05
12	0	1	-1	27	26.96	27.00	0.04	0.00
13	-1	1	0	27	26.96	27.00	0.04	0.00
14	-1	0	-1	21	21.04	21.00	-0.04	0.00
15	0	-1	-1	27	26.96	27.01	0.04	0.01

Table 4 Test of Significance for Regression Coefficient

Source	DF	Seq. SS	Contribution%	Adj. SS	Adj. MS	F-Value	P-Value
X_1	1	288.000	54.57	288.000	288.000	3744.00	0.000
X_3	1	0.000	0.00	0.000	0.000	0.00	1.000
X_1^2	1	117.376	22.24	115.522	115.522	1501.79	0.000
X_3^2	1	0.665	0.13	0.665	0.665	8.64	0.016
X_1X_3	1	121.000	22.93	121.000	121.000	1573.00	0.000

Where: *DF* Degree of Freedom, *Seq. SS* Sequential Sum of Square, *Adj. SS* Adjusted Sum of Square, *Adj. MS* Adjusted Mean Square, *F* Fischer, *P* Probability

Table 5 Analysis of Variance (ANOVA) of Regression Equation

Source	DF	Seq. SS	Contribution%	Adj. SS	Adj. MS	F-Value	P-Value
Model	5	527.041	99.87	527.041	105.408	1370.31	0.00
Lack-of-Fit	7	0.026	0.00	0.026	0.004	0.01	1.00
Pure Error	2	0.667	0.13	0.667	0.333		
Total	14	527.733	100.00				
R^2= 99.87%, R^2 (adjusted)= 99.80%, R^2 (predicted)= 99.77%							

Where: *DF* Degree of Freedom, *Seq. SS* Sequential Sum of Square, *Adj. SS* Adjusted Sum of Square, *Adj. MS* Adjusted Mean Square, *F* Fischer, *P* Probability

The significance of regression was evaluated by the *F* value and the *p* values using Fischer's and null-hypothesis tests; where the *F* value predicts the quality of the entire model considering all design factors at a time, whereas the *p* value denotes the probability of the factors having very little or insignificant effect on the response. A higher *F* value signifies a better fit of the RSM model to the experimental data [61]. According to [62], an *F* value along with low *p* value indicates a high significance of the regression model. Nevertheless, the *p* value should be lower than 0.05 for the model to be statistically significant [63]. Based on these reports, the regression model found in this study was highly significant, as is evident by the large *F* value = 1370.31 and the low *p* values = 0.000, respectively.

To test the fit of the model equation, the regression model was established using R^2 as a measure of how much variability in the observed response values can be explained by the experimental factors and their interactions [64]. The R^2 value is always between 0 and 1 [37, 65]. However, for creating a good-fit model, it was recommended that R^2 should not be less than 80% [66]. The results in Table 7 indicate an R^2 value of 99.87% which leaves only 0.13% of the variability in the observed response values to be not explainable by the model and indicates that an unexplainable total variation could be caused by other factors, which were not included in the model.

The graphical depiction provides a method to visualize the relationship between the response and the experimental levels of each variable on the one side, and the type of interactions between the test variables, on the other, which allows for deducing the optimum conditions. The contour and surface plots for oil extraction from *T. peruviana* seed oil under the feasible optimum conditions for Minitab RSM and ANN are shown in Fig. 1a-f A plot of the linear correlation between the experimental yields and the predicted value for oil extraction by ANN is presented in Fig. 2, indicating that the models are adequate without any violation of independence or constant assumption. The accuracy of the models obtained from MINITAB RSM and ANN was determined by comparing the values of R^2. The result depicts that both optimization tools allowed good predictions to be achieved due to the value of R^2 (of 99.870 and 99.936% for RSM and ANN, respectively). Therefore, for the extraction of *T. peruviana* oilseed, ANN showed a clear superiority over Minitab RSM due to the R^2 value and the predicted optimum yield.

Properties of the *T. peruviana* seed oil

The properties of the *T. peruviana* seed oil were determined according to the standard methods, and the results are shown in Table 7. The oil obtained was liquid, yellowish in color with a moisture content of 0.0131% and a specific gravity of 0.8984 g/cm^3, at a density of 0.774 g/cm^3. The obtained acid and FFA values of 3.8048 and 1.9024 point to a good resistance of the oil to hydrolysis, whereby the abovementioned values are in close agreement with the results obtained by other researchers. The peroxide value measures the content of hydroperoxides in the oil [67], and its low value (23.8 meq. O_2/kg) indicated a high resistance to oxidation. A high saponification value of 157.5025 mg KOH/g and an iodine value of 97.60 gI_2/100 g pointed to both a low concentration of triglycerides and an oil containing a substantial level of unsaturation. The high HHV (57.612 MJ/kg) was associated with the latent heat of

Table 6 Regression coefficients and significance of response surface quadratic

Term	Effect	Coefficient	Standard Error Coefficient	95% CI Low	95% CI High	P-Value	T-Value	VIF
Constant	-	27.385	0.1330	27.083	27.686	0.000	205.54	-
X_1	12.000	6.000	0.0981	5.7782	6.2218	0.000	61.19	1.00
X_3	0.000	0.000	0.0981	-0.2218	0.2218	1.000	0.00	1.00
X_1^2	11.154	5.577	0.1440	5.251	5.902	0.000	38.75	1.01
X_3^2	-0.846	-0.423	0.1440	-0.749	-0.098	0.016	-2.94	1.01
X_1X_3	-11.000	-5.500	0.1390	-5.814	-5.186	0.000	-39.66	1.00

Table 7 Physical, Chemical and Other properties of *T. peruviana* seedoil extracted

Parameters	Yellow oleander seed oil
Physical properties	
Physical state at room temperature	Yellowish in colour
Moisture content (%)	0.0131
Specific gravity	0.8984
Mean Molecular mass	973.87
Chemical properties	
%FFA (as oleic acid)	1.9024
Acid value (mg KOH/g oil)	3.8048
Saponification value (mg KOH/g oil)	57.5025
Iodine value (g I_2/100g oil)	97.600
Peroxide value (meq O_2/kg oil)	23.800
Higher heating value (MJ/kg)	47.056
Other properties	
Cetane number	140.998
API	26.00
Diesel index	181.94
Aniline point (^{o}F)	699.77

vaporization of water in the combustion products. Both fuel properties and cetane number (CN) are not only a measure of the fuel's ignition delay and combustion quality, but also of its biodiesel standard with a minimum of 40 [68, 69] BIS HIE. The high value (58.99) obtained in this study shows that the oil has high fuel potential. The API and diesel index of *T. peruviana* seed oil was less than that of AGO but are comparable with other vegetable oils earlier reported by [70, 71].

Gas chromatographic analysis of *T. peruviana* seed oil

GC-MS is one of the techniques which allows for the identification of the constituents of volatile matter, long and branched chain hydrocarbons, alcoholic acids, esters and other components [72]. The results pertaining to the analysis leaded to the identification of the number of compounds from the GC fractions of *T. peruviana* seed oil. The analysis had proved that the oil contained linoleic (37.91%), oleic (32.82%), linolenic (12.33%), palmitic (9.52%), stearic (7.02%), and other (0.40%) acids. It was observed that it also contained a substantial level of unsaturation (70.73%), which implies a low saponification value and a high iodine value (Table 8).

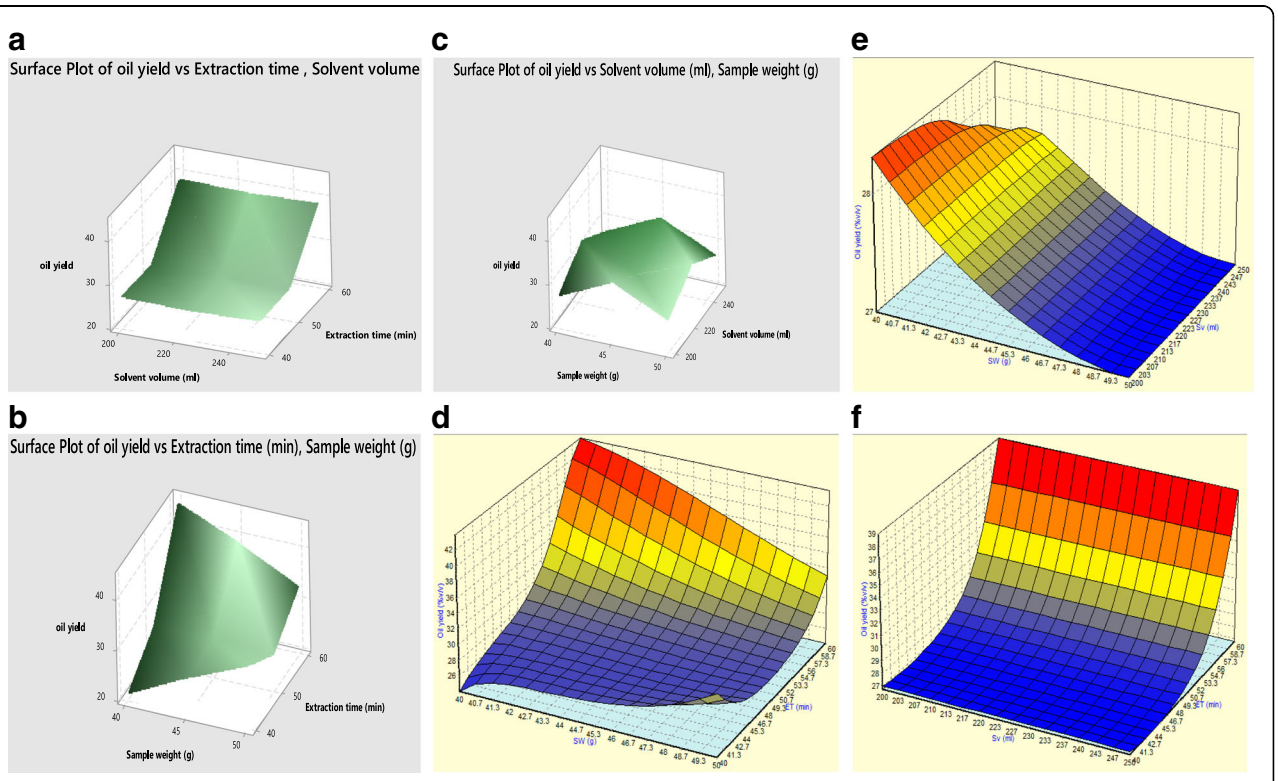

Fig. 1 3D surface plot of MRS showing interactive effect of **a** solvent volume and extraction time on *T. peruviana* oil yield % (v/v); **b** extraction time and sample weight on *T. peruviana* oil yield % (v/v); **c** solvent volume and sample weight on *T. peruviana* oil yield % (v/v). **d** 3D surface plot of ANN showing interactive effect of sample weight (SW) and extraction time (ET) on *T. peruviana* oil yield % (v/v); **e** sample weight (SW) and solvent volume (SV) on *T. peruviana* oil yield % (v/v); **f** solvent volume (SV) and extraction time (ET) on *T. peruviana* oil yield % (v/v)

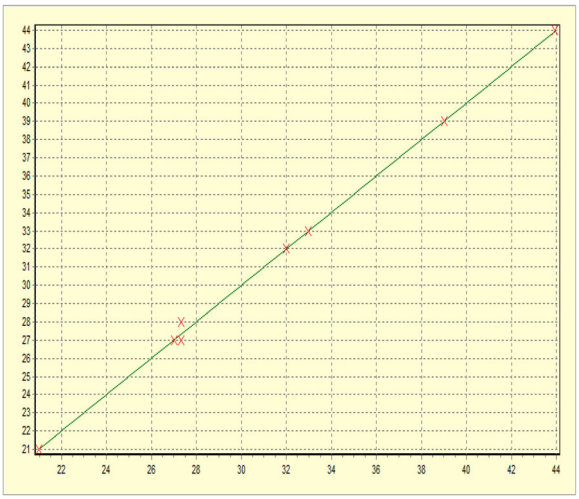

Fig. 2 Linear correlation between oil yield %(v/v) (vertical axis) and ANN value %(v/v) (horizontal)

EDXRF catalyst composition analysis

Table 9 demonstrates the results of the elemental analysis using an EDXRF spectrophotometer (EDX3600B) for the calcinated grounded BPSM samples A, B, C, D, E, F, G, and H in a furnace at 700 °C. Samples A, D, E, and H were pre-soaked in methanol for 10 min, while samples B, C, F, and G were not pre-soaked. Samples A and B were calcinated for 4 h, samples C and D for 5 h, samples E and F for 6 h, and samples G and H for 7 h, respectively. From Table 9 and Fig. 3 (the corresponding spectrum), it was evident that sample D had the highest base content of 58.48%, while sample B had the lowest amount of base at 54.73%. Therefore, pre-soaking pulverized BPSM in methanol and its calcination increased the elemental composition of base present in the sample.

Esterification process catalyzed *T. peruviana* seed oil acid

Table 10 presents the esterification conditions along with their corresponding acid and FFA values. The optimum condition obtained for the pre-treatment of 75.00 ml of *T. peruviana* seed oil was established at a methanol/acid volumetric ratio of 12 ml and a methanol volume of 75 ml; the acid value obtained under these conditions was 0.7896 mg $KOHg^{-1}$ with a corresponding FFA value of 0.3948 mg $KOHg^{-1}$ that was required for a successful transesterification of oil to biodiesel [35, 73].

Table 8 Percentage prevailing compound from gas chromatography analysis of *T. peruviana* oil

S/N	Acids Compounds	Percentage (%)
1	Linoleic	37.91
2	Oleic	32.82
3	Linolenic	12.33
4	Palmitic	9.52
5	Stearic	7.02
6	Other	0.40
	Total	100
7	*Unsaturation*	70.73
8	*Saturation*	28.87

Transesterification process catalyzed by *T. peruviana* seed oil base

Table 11 illustrates the coded factors, the experimental results, the predicted values, as well as the residual values obtained for both MRS and ANN. The maximum obtained experimental *T. peruviana* biodiesel yield reached 86.00% (*v*/*v*) at a catalyst amount of 4 g: X_1, a reaction time of 70 min: X_2 and a methanol/oil ratio of 0.1: X_3. The optimum predicted yield achieved via MRS was 86.40% (*v*/*v*) at a catalyst amount of 3.8 g: X_1, a reaction time of 69 min: a X_2 and methanol/oil ratio of 0.11: X_3; ANN also predicted the optimum value of 86.80% (*v*/*v*) at a catalyst amount of 3.79 g: X_1, a reaction time of 68.50 min: and X_2 a methanol/oil ratio of 0.10: X_3. These predicted yields were validated by carrying out three experiments, and average yields of 85.70% (*v*/*v*) and 85.98% (*v*/*v*) were obtained for MRS and ANN, respectively. Table 12 listed the results of the test of significance for every regression coefficient. The results indicated that the p values ($p < 0.05$) of the model terms were significant. Considering this case, X_1, X_2, X_3,X_2^2, and X_3^2 of linear and quadratic terms were found to be significant at a 95% confidence level. However, with reference to the large F values and the corresponding low p values, the catalyst amount X_1 (F value = 13,467.00) was the most significant variable followed by the methanol/oil ratio: X_3 (F value = 4563.00) followed by the reaction time: X_2 (F value = 108.00); however, the quadratic term X_3^2 with the F value = 11,215.38 was more significant than X_2^2 with an F value = 10,722.46. To minimize the error, all the coefficients were taken into consideration in the design. The ANOVA of regression is represented in Table 13. The model F value = 4835.25 with a low $p < 0.0001$ implied a high significance for the regression model [74]. The final equation in terms of the coded factors for the MRS quadratic model is expressed by Eq. (10).

$$\begin{aligned} Y\%(v/v) = {} & 581.63 - 5.438X_1 - 13.100X_2 - 1417.5X_3 \\ & + 0.0625X_1^2 + 0.11000X_2^2 \\ & + 4500.0X_3^2 \\ & + 0.01250X_1X_2 - 0.500X_2X_3 \end{aligned} \tag{10}$$

All the linear terms (X_1, X_2, X_3) and cross-product terms (X_2X_3) had negative effects on the *T. peruviana* biodiesel yield (Y), while the cross-product term (X_1X_2)

Table 9: Results of calcinated samples analysis using EDXRF Spectrophotometer

Calcinated samples	$A_{(pre\text{-}soaked)}$	B	C	$D_{(pre\text{-}soaked)}$	$E_{(pre\text{-}soaked)}$	F	G	$H_{(pre\text{-}soaked)}$
Duration (hrs)	4	4	5	5	6	6	7	7
% Potassium	56.19	54.73	58.14	58.48	56.94	57.89	55.16	56.08

and all surface quadratic (X_1^2, X_2^2, X_3^2) model terms had positive effects on the *T. peruviana* biodiesel yield. The ANOVA results of the regression equation are presented in Table 14. The significance of each coefficient in the experimental model was also determined by T value and P value. A high T test value and a low P value indicate a high significance [75, 76]. Student's t test of each coefficient of the model showed that the linear coefficient (X_2), the quadratic term $({X_1}^2, {X_2}^2, {X_3}^2)$ and the cross product (X_1X_2) have significant effects $(P > |T| < 0.05)$ on

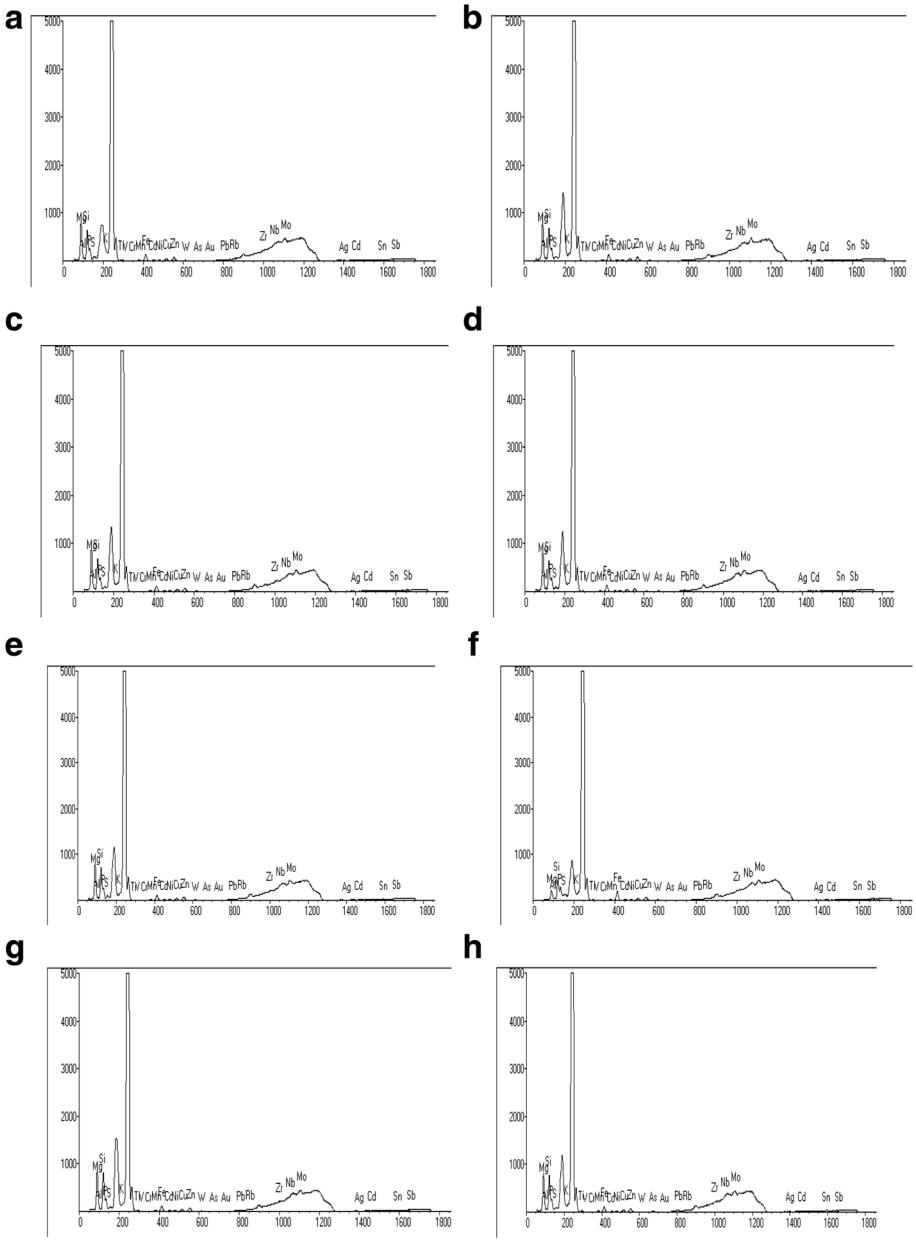

Fig. 3 a-**h** EDXRF Spectrophotometer spectrum curves for catalyst samples

Table 10 Esterification condition with corresponding Acid and FFA values

Methanol/acid volume ratio	Acid value ($mgKOHg^{-1}$)	FFA ($mgKOHg^{-1}$)
-	3.8048	1.9024
3.00	2.6920	1.8460
6.00	2.2792	1.1396
9.00	1.3536	0.6768
12.00	0.7896	0.3948

T. peruviana biodiesel (Y). All other terms displayed are not significant. The low values of standard error observed in the intercept and all the model terms demonstrated that the regression model fit the data, and the prediction was good [37]. The variance inflation factor (VIF) obtained in this study illustrates that the centre points are orthogonal to all other factors in the model.

The 3D surface plots are graphical representations of the regression equation for the optimization of the reaction variables, and they are presented in Fig. 4a-f. The curvatures' nature as plotted by MRS of the two- and three-dimensional surfaces in Fig. 4a-c shows an interactive effect among the variable selected and *T. peruviana* biodiesel yield. Furthermore, the curvatures' nature of the three-dimensional surfaces as plotted by ANN (Fig. 4d-f) suggested mutual interaction among the selected variables. The importance level of the selected variables considered for *T. peruviana* biodiesel by ANN is illustrated in Fig. 5, and it was evident that catalyst amount X_1 was the most important variable factor (53.54%) followed by methanol/oil ratio (41.31%) and reaction time (5.15%), respectively.

Furthermore, the accuracy of the models achieved by MRS and ANN based on R^2 was evaluated. The results depict that both optimization tools gave good predictions of R^2 (MRS: R^2 = 99.98% and ANN: R^2 = 99.97%).

Effects of T. peruviana oilseed (feedstock) on the properties of biodiesel

From the results, it was observed that *T. peruviana* seed is rich in oil, and its properties such as the low moisture content, the specific gravity, the diesel index, the cetane number, and the higher heating value make it suitable for biodiesel production. Furthermore, the high free fatty acid inherent in the oil points to its non-edibility and high unsaturation. Hence, the oil from *T. peruviana* seed is a good feedstock for environmental friendly biodiesel production.

Effects of the catalyst (BPSM) on the properties of biodiesel production

The results of an elemental analysis using an EDXRF spectrophotometer (EDX3600B) for calcined BPSM reveal that sample D has the highest base content of 58.48% (Table 9). Fig. 3a-h shows the corresponding spectrum with the relative abundance of the elements present. It was observed that various metals both in micro and in macro found in the calcinated sample, ranged from magnesium (Mg) to antimony (Sb). However, potassium demonstrated the highest relative abundance in

Table 11 Coded experimental design results, *T. peruviana* biodiesel yield, predicted values by MRS and ANN and the residual values for the transesterification process

Run	X_1	X_2	X_3	Biodiesel yield %(v/v)	Predicted		Residual	
					MRS	ANN	MRS	ANN
1	1	0	1	56.00	56.25	55.93	-0.25	0.07
2	1	-1	0	60.00	59.88	60.63	0.13	0.63
3	0	1	-1	86.00	86.13	86.19	-0.12	0.19
4	-1	1	0	78.00	78.13	78.02	-0.12	0.02
5	0	0	0	58.00	58.00	58.02	0.00	0.02
6	0	1	1	76.00	75.88	75.98	0.13	0.02
7	-1	-1	0	77.00	77.13	76.99	-0.12	0.02
8	0	0	0	58.00	58.00	58.02	0.00	0.02
9	1	0	-1	66.00	66.00	66.08	0.00	0.08
10	0	0	0	58.00	58.00	58.02	0.00	0.02
11	-1	0	-1	83.00	82.75	82.99	0.25	0.01
12	0	-1	1	75.00	74.88	75.02	0.13	0.02
13	1	1	0	62.00	61.88	61.34	0.13	0.66
14	-1	0	1	73.00	73.00	73.00	0.00	0.00
15	0	-1	-1	84.00	84.13	83.81	-0.12	0.19

Table 12 Test of significance for every regression coefficient

Source	DF	Seq. SS	Contribution (%)	Adj. SS	Adj. MS	F-Value	P-Value
X_1	1	561.13	34.81	561.13	561.13	13467.00	0.00
X_2	1	4.50	0.28	4.50	4.50	108.00	0.00
X_3	1	190.13	11.79	190.13	190.13	4563.00	0.00
X_1^2	1	6.70	0.42	0.23	0.23	5.54	0.06
X_2^2	1	381.50	23.67	446.77	446.77	10722.46	0.00
X_3^2	1	467.31	28.99	467.31	467.31	11215.38	0.00
X_1X_2	1	0.25	0.02	0.25	0.25	6.00	0.05
X_2X_3	1	0.25	0.02	0.25	0.25	6.00	0.05

Where: *DF* Degree of Freedom, *Seq. SS* Sequential Sum of Square, *Adj. SS* Adjusted Sum of Square, *Adj. MS* Adjusted Mean Square, *F* Fischer, *P* Probability

all the samples, while titanium (T), vanadium (V), chromium (Cr), manganese (Mn), arsenic (As), gold (Au), and tin (Sn) showed the lowest relative abundance among others. The observation from this study, which implies that K is the major active component responsible for the activity of the catalyst in BPSM synthesis can be supported by the findings reported by Betiku et al. [35], where K with 59.3% has the highest concentration among other elements such as calcium, magnesium, phosphorous, and others present in calcinated cocoa pod husk used in biodiesel production from *Azadirachta indica* seed oil. In this study, BPSM reached the highest concentration of 58.48% at a calcination time of 5 h for the peak value. This observation could not only be caused by its macro mineral nature and maintenance of osmotic pressure, but also by its cell size in the plant, which influenced the photosynthesis and energy production found in potassium. The calcination also resulted in the sintering of small mineral aggregates and agglomerated particles, which explained the spongy nature. It had been reported that calcination always prevented leaching of potassium and may increase the reusability of catalyst during biodiesel production [35]. These effects were associated with higher heating values and cetane numbers of biodiesel.

Quality characterization of *T. peruviana* biodiesel compared to other studies using the ASTM D6751 and EN 14214 standards, for example

The quality of biodiesel obtained after the transesterification process was evaluated by subjecting the content and compositions of *T. peruviana* biodiesel to physicochemical analysis. The results obtained were compared with other published articles and also with ASTM D6751 and EN 14214 standards (Table 15). At room temperature, the *T. peruviana* biodiesel produced was liquid, brownish in color with a moisture content of 0.004. Observation from other researchers published showed no value, but the ASTM D6751 allowed to achieve a value < 0.03 and EN 14214 of 0.02. The density of 0.816 g/cm^3 was in reasonable agreement with other works and within the range specified by ASTM D6751 and EN 14214. The biodiesel acid value achieved was 0.508 mg of KOH/g, which was higher than in other published work but within the prescribed ASTM D6751 (< 0.8) and EN 14214 (0.5 max). The iodine value of *T. peruviana* biodiesel of 73.20 gI_2/100 g oil obtained was above the value recorded in [43] but lower than the result published in [35], and below the maximum limit of 120, prescribed by EN 14214. The saponification value, which is the number of milligrams of KOH required to neutralize the fatty acids resulting from the complete hydrolysis of 1 g of fat or oil, gives an indication of the nature of the fatty acids of oil and thus dependent on the average molecular weight of the fatty acids constituent of the oil. The value obtained was 126.648 mg KOH/g oil, but no value was recorded by other researchers in the previously published works. The higher heating value (HHV) determined for the biodiesel in this study was 45.335 MJ/kg, which takes into account the latent heat of vaporization of water in the combustion products. The cetane number, which is a measure of the fuel's ignition delay and combustion quality of the oil/fuel;

Table 13 Analysis of Variance (ANOVA) of regression equation

Source	DF	Seq.SS	Contribution (%)	Adj. SS	Adj. MS	F-Value	P-Value
Model	8	1611.75	99.98	1611.75	201.47	4835.25	0.00
Lack-of-Fit	4	0.25	0.02	0.25	0.06	*	*
Pure Error	2	0.00	0.00	0.00	0.00		
Total	14	1612.00	100.00				

R^2 = 99.98% , R^2(adj.) = 99.96%, R^2 (pred.) = 99.84%

Where: *DF* Degree of Freedom, *Seq. SS* Sequential Sum of Square, *Adj. SS* Adjusted Sum of Square, *Adj. MS* Adjusted Mean Square, *F* Fischer, *P* Probability

Table 14 ANOVA for response surface quadratic model for intercept

Term	Coefficient	Standard Error Coefficient	95% CI Low	95% CI High	T- Value	P- Value	VIF
Constant	58.00	0.1180	57.712	58.288	492.15	0.000	-
X_1	-8.375	0.0722	-8.5516	-8.1984	-116.05	0.000	1.00
X_2	0.75	0.0722	0.5734	0.9266	10.39	0.000	1.00
X_3	-4.875	0.0722	-5.0516	-4.6984	-67.55	0.000	1.00
X_1^2	0.25	0.1060	-0.01	0.51	2.35	0.057	1.01
X_2^2	11.00	0.1060	10.74	11.26	103.55	0.000	1.01
X_3^2	11.25	0.1060	10.99	11.51	105.9	0.000	1.01
X_1 X_2	0.25	0.1020	0.00	0.50	2.45	0.050	1.00
X_2 X_3	-0.25	0.1020	-0.50	0.00	-2.45	0.050	1.00

as the higher the cetane number and the shorter the delay interval is, the greater the combustibility will be. Fuels with a low cetane number are difficult to start, hence producing smoke. The standard minimum specification value of the cetane number for biodiesel is within the range of 47–51 (ASTM D6751 and EN 14214). The value obtained by Betiku [35] was high (123.251), but the value obtained in this study reached only 72.926. This may be attributed to the nature of the catalyst used during the transesterification process. Other properties, such as aniline point, diesel index, and API of the *T. peruviana* biodiesel were also evaluated and recorded. The *T. peruviana* diesel index (87.397) and aniline point (372.155) obtained were higher than that of neat diesel; the API (23.484) was lower

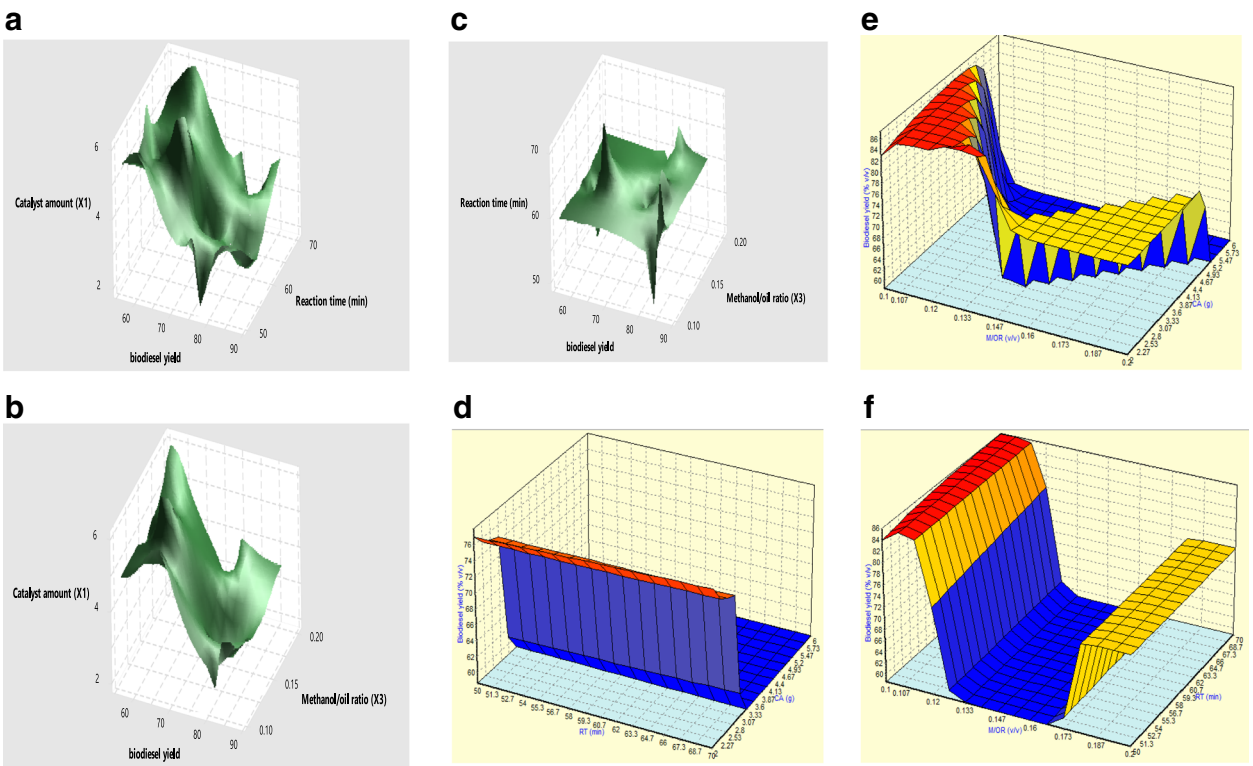

Fig. 4 **a** 3D surface plot of MRS showing interactive effect of catalyst amount and reaction time on biodiesel yield % (v/v); **b** catalyst amount and methanol/oil ratio on biodiesel yield % (v/v); **c** reaction time and methanol/oil ratio on biodiesel yield % (v/v). **d** 3D surface plot of ANN showing interactive effect of catalyst amount and reaction time on biodiesel yield % (v/v); **e** catalyst amount and methanol/oil ratio on biodiesel yield % (v/v); **f** reaction time and methanol/oil ratio on biodiesel yield % (v/v)

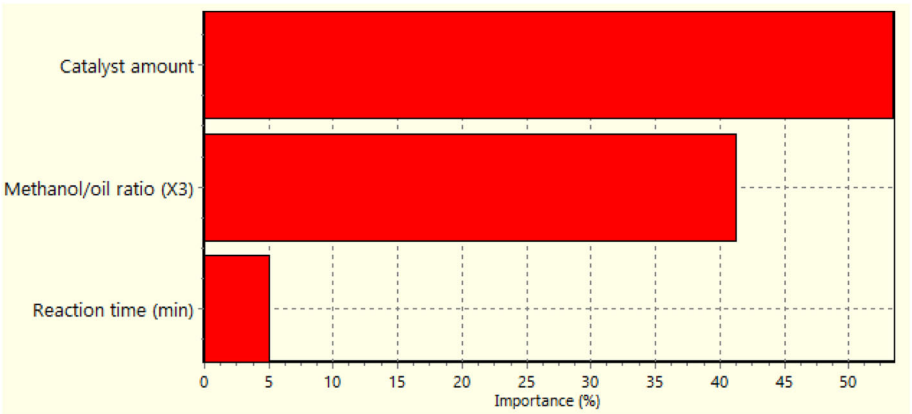

Fig. 5 Importance of selected variables considered on the percentage biodiesel production

compared to that of neat diesel, but was comparable to that which was obtained by other researchers using different seed oil biodiesel [70, 71].

Comparing the fuel properties of *T. peruviana* oil with biodiesel

The suitability of the *T. peruviana* biodiesel in I.C. engines was proved by the evaluation of the fuel properties. The results obtained are displayed in Table 16. It was observed from the parent oil (*T. peruviana* seed oil) to the biodiesel that the moisture content was reduced by 66.4%, which showed very low moisture content of the biodiesel which was required in order to prevent or eliminate an engine knockout effect. The specific gravity increased by 1.6%, which pointed to a significant increase in heat of vaporization and accelerated ignition when combustion occurred. The iodine value was reduced by 24.69%, which implies that the biodiesel produced has a certain level of unsaturation [77, 78], whereas the acid value was reduced by 71.8%, indicating that the biodiesel has a long shelf life [79, 80–83]. The saponification value was reduced by 30.86%, suggesting a low concentration of triglycerides in biodiesel, while the density increased by 5.15%. However, the high heating value further increased by 0.54%, when converted to biodiesel, and the cetane number also increased by 19.11%, which accounts for its greater combustion process ability, as a fuel with low a cetane number produces smoke, when used in an engine [80]. Other additional fuel properties, such as the aniline point increased by 29.68%, and the diesel index increased by 22.15%. The API decreased by 10.71% from oil to biodiesel. These results confirmed

Table 15: Qualities of *T. peruviana* biodiesel as compared with other researched work

Properties	[81]	[44]	[31]	[82]	[83]	[35]	ASTM D6751	EN 14214	This study
Density (15 °C, g/cm^3)	0.839	0.875	0.86	0.87	0.866	0.887	0.84	0.86-0.90	0.816
Acid value (mg KOH/g)	-	0.057	0.3	0.2	-	0.46	<0.80	0.5 max.	0.508
Free fatty acid (mg KOH/g)	-	-	-	-	-	-	<0.40	0.25 max.	0.254
Iodine value (g I_2 /100 g)	-	69.9	-	-	-	-	-	120 max.	73.20
Saponification value (mg KOH/g)	-	-	-	-	-	-	-	-	26.648
Kinematic viscosity (40 °C, mm^2/s)	4.2	4.33	5.17	4.5	5.1	6	-	-	3.92
Cetane number	47	61.5	-	54.2	-	123.25	47 min.	51 min.	234.58
Moisture content (wt. %)	-	-	-	-	-	-	<0.03	0.02	0.0044
Specific gravity	-	-	-	-	-	-	0.86-0.90	0.85	0.913
Centane index	-	62.9	-	-	58.97	-	-	-	-
Mean Molecular Mass	-	-	-	-	-	-	-	-	2101
Higher Heating Value (MJ/kg)	-	-	-	-	-	-	-	-	47.312
API	-	-	-	-	-	-	36.95	-	23.484
Diesel index	-	-	-	-	-	-	50.4	-	311.918
Aniline point (°F)	-	-	-	-	-	-	331	-	1328.24

Table 16: Comparing the fuel properties of *T. peruviana* oil and biodiesel

Parameters	*T. peruviana* oil	*T. peruviana* biodiesel
Density (g/cm^3)	0.774	0.816
Moisture content (%)	0.0131	0.0044
Specific gravity	0.8984	0.913
Mean Molecular mass	973.87	2101
Acid value (mg KOH/g oil)	3.8048	0.508
Saponification value (mg KOH/g oil)	57.5025	26.648
Iodine value (g I_2/100g oil)	97.6	73.2
Peroxide value (meq. O_2/kg oil)	23.8	-
Higher heating value (MJ/kg)	47.056	47.312
Cetane number	140.998	234.58
API	26	23.484
Diesel index	181.94	311.918
Aniline point (^{o}F)	699.77	1328.239

that the produced *T. peruviana* biodiesel could serve as an alternative to the conventional diesel and its blends could improve fuel properties.

Conclusions

In conclusion, in this study, the pre-soaked calcinated BPSM was proved to serve as an alternative catalyst for biodiesel production from *T. peruviana* oil. *T. peruviana* seed was found to be rich in oil with an average yield of 44.00% (*v*/*v*), and the oil was highly unsaturated with a high FFA. The maximum experimental biodiesel yield obtained was 86.00% at a catalyst amount of 4 g, a reaction time of 70 min, and a methanol/oil ratio of 0.1(*v*/*v*). The properties of the *T. peruviana* biodiesel, as described in other earlier reports using the same feedstock with different catalysts as well as compared with ASTM D6751 and EN 14214, indicated that the produced biodiesel had properties which agreed to those reported in the literature. Hence, it can be concluded that *T. peruviana* biodiesel produced in the case where a blend could serve as an alternative to conventional diesel is environmentally friendly.

Abbreviations

AEOE: Aqueous enzymatic oil extraction; ANN: Artificial neural network; ANOVA: Analysis of variance; ASTM: American Society of Testing and Material; BPSM: Brette Pearl Spar Mable; CN: Cetane number; EDXRF: Electron diffractometer X-ray fluorescence; FAME: Fatty acid methyl ester; FFA: Free fatty acid; GC-MS: Gas chromatographic mass spectrometer; GR&R: Gage repeatability and productivity; HHV: Higher heating value; I.C engine: Internal combustion engine; IV: Iodine value; MRS: Minitab response surface; PV: Peroxide value; RSM: Response surface methodology; SV: Saponification value; VIF: Variance inflation factor

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Availability of data and materials

All data are included in the article.
Raw data are not applicable.

Declarations

That the article is original, has not already been published in a journal, and is not currently under consideration by another journal.

Authors' contributions

TFA was involved in the research work and prepared the manuscript. BEO designed the experiment, supervised the work, and also carried out the extraction part of the work, while OMO performed the elemental calcination part of the work. Finally, MAI carried out the biodiesel production part and the analysis of biodiesel. All authors worked together to achieve the optimum outcome of this research work. All authors read and approved the final manuscript.

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