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Optimization for Enhanced Phase Separation and Purification of Tropical Almond Oil Biodiesel System Using Response Surface Methodology

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Abstract

In the present study, the extract phase components of tropical almond oil biodiesel ternary mixture were investigated using response surface methodology under different process conditions. The parameters employed were temperature (°C), withdrawal time (min), and phase component composition (mg/mL). Second-order polynomial regression model was used to predict the response and determine the variables at which optimized outputs were obtained thus predicting the best model for enhancing the extract phase component separation and purification. Firstly, experiments were performed according to central composite design. The model predicted is quadratic with optimal temperatures and time intervals for extract phase separation and purification achieved at: 60 °C, 18 mins, 60 °C, 6 mins, and 30 °C, 14 for tropical almond oil biodiesel, methanol, and glycerol components respectively. The prediction measurements at the optimal conditions were: coefficient of determination, R^2 = 0.98126, adjusted coefficient of determination, adj. R^2 = 0.9510, root mean square error (RMSE) = 0.016529, sum of square error (SSE) = 0.037704. This study provides a green approach for enhanced phase separation and purification of biodiesel to meet future renewable energy demand.

1. Introduction

Currently, biodiesel offers the most encouraging source of renewable energy aiming to replace fossil diesel fuel as a result of some inherent physico-chemical properties [1]. Besides, specific fuel properties of petrol-diesel and biodiesel are comparable, and therefore, biodiesel is useable with minor modifications on diesel engines [2]. Biodiesel is biodegradable and has low levels of toxicity. It is environmentally friendly with almost zero emissions of CO, CO_2, NO_x, SO_x levels when compared to fossil diesel. It has potential benefits and significant improvements to rural economic lives [3]. Studies reveal that specific fuel properties of biodiesel enhance its use as combustion fuel in diesel engines [4]. It has encouraged the development of suitable models for the study of phenomena occurring in the application of biodiesel in engines. Similarly, optimization of pertinent parameters in the determination of fuel properties of biodiesel and purification using research laboratory test procedures are time-consuming and grossly inefficient.

Generally, three main steps are required to convert vegetable oils to fatty methyl esters (biodiesel). The first is pre-treatment of the vegetable oil to remove free fatty acids that could interfere with the appropriate reaction mechanism. The second is the reactive step known as the transesterification reaction, and the third is a sequence of purification procedures to obtain the final product that must conform to accepted international standards. Transesterification reaction can generate pure fatty methyl esters. However, the purification step is usually required to separate esters obtained from glycerol, excess alcoholic reagents, and other contaminants introduced into the process. The purity and quality of biodiesel have influences on its fuel properties. Therefore, the final biodiesel product should be free from water, alcohol, glycerol, and catalyst. The washing of produced biodiesel using different media is to remove residues of alcohol, glycerol, catalyst and soap [5, 6]. It is achieved without any defined prediction and optimization process for the purification using the different washing media. Thus, the phase separation and purification steps in biodiesel production cannot be overemphasized. The level of purity of any biodiesel has effects on its final fuel properties [7].

The response surface methodology (RSM) model is a powerful tool whose application and benefits cut across several fields of human endeavor. RSM is used to determine the optimum conditions for production, separation, and purification processes in biodiesel systems [8]. To achieve optimum purification of biodiesel after the production and separation, process variable which includes biodiesel phase components composition (biodiesel, methanol, and glycerol), time interval and temperature can be utilized simultaneously in RSM using central composite design (CCD) [9]. With RSM, the number of experimental runs will reduce, resulting in statistically acceptable results based on adequate evidence presented [10]. Several studies on the use of RSM in the optimization of the transesterification process of vegetable oil to biodiesel exist [11, 12, 13].

One study investigated the optimization of the castor seed oil extraction process by response surface methodology (RSM) [14]. The authors employed the Taguchi design to determine investigated process parameters. The RSM gave the best process parameter in terms of temperature and pressure for better oil yield using mechanical extraction. Another study [15] investigated the optimization of fatty methyl ester production using RSM and artificial neural network (ANN) applying central composite design (CCD) optimization process parameter. The optimization method provided estimation for biodiesel yield in the batch transesterification process with no reference to the phase separation and purification process. The response model was used to predict the best model for maximizing biodiesel production. The optimization of biodiesel production from selected waste oils using response surface methodology was investigated by other researchers [10]. Their findings revealed different response models for biodiesel yields from the selected waste oil. The influence of the model on phase separation and purification was not investigated. Some other authors [16] discussed the application of modeling techniques for predicting and optimization of the biodiesel production processes. They highlighted modern trends in biodiesel modeling process while stating possible scopes and a range of applications. The role of the phase separation and purification in biodiesel production process was however excluded. Some researchers [17] employed response surface methodology to optimize the dilution level and agitation time for castor oil extraction based on the percentage recovery of the oil. A second-degree polynomial regression model was obtained based on the optimized parameters. In these studies, effect of optimization on the phase separation and purification of the different biodiesel systems were never investigated.

Tropical almond tree (*terminalia catappa*) is a large tropical tree that grows mainly in tropical regions of the world. In Nigeria, it is found mostly in the north-central and southern parts of the country. It provides shade to homes, offices, and surroundings. Its fruit consist of the kernel, hull, and seed coat. The fruit litters the environment as waste. Thus, their use as feedstock for biodiesel production would also serve as veritable resource utilization and waste disposal option [18].

In this research, RSM model is used to predict and optimize the phase separation and purification of tropical almond oil biodiesel system from which contains a mixture of tropical almond oil biodiesel, glycerol, and methanol. It is required to ascertain the quality and purity levels of the final biodiesel mixture. Thus, enhancing the viability of the industrial application of the purification process based on knowledge of optimum conditions for the operating variables. The use of RSM model approach has not been undertaken anywhere for biodiesel phase separation, and purification process. It has not been applied for the purification process of the tropical almond oil biodiesel system. This research is undertaken to fill this existing knowledge gap.

2. Materials and methods

2.1. Materials

The materials used in this research are tropical almond seeds obtained from agrarian villages in Ankpa, Kogi State, Nigeria; tropical almond seed oil extracted mechanically; tropical almond oil biodiesel manufactured by an alkaline transesterification reaction. The chemicals used included glycerol (99% -100%), methanol (99.5%) and potassium hydroxide pellets (85%). They were products of Sigma-Aldrich, U.K and J.T. Baker, USA. The chemicals were of analytical grade and used as purchased.

2.2 Methods

2.2.1 Analysis of tropical almond oil and biodiesel

The tropical almond oil and biodiesel obtained were subjected to fatty acid analysis. The fatty acid composition of the oil and biodiesel were determined by GC method following ASTM D6584, EN14214, and EN14105 using a Thermo Scientific Trace GC Ultra AS 3000 Auto-Sampler gas chromatography/mass spectrometer connected to a flame ionization detector (FID). The criteria were both qualitative and quantitative for the analysis of the oil and biodiesel produced as specified in the ASTM and EN standards. The physicochemical properties of the oil and biodiesel which included the fatty acid composition and FAME analysis using GC have previously been reported [19].

2.2.2 Determination of phase components composition

Processes employed in the analysis of extract phase mixture composition of tropical almond oil biodiesel/methanol/glycerol system

Burette, mechanical agitator, analytical weighing balance, graduated pipette, thermostatic water-bath, conical flasks, stopwatch, beakers, and gas chromatograph-mass spectroscopy/flame ionization detector (GCMS/FID) for analysing the composition of the mixture were the equipment employed in the extract phase experiments. Cloud point titration at the different temperatures and time intervals were used to determine the tie line data for the tropical almond biodiesel ternary system. This same procedure was employed by other authors [20, 21].

2.2.3 Design of experiments and statistical analysis using RSM

The optimization modeling approach of biodiesel systems involves several challenges among that include phase separation of products, mass transfer from the reaction, chemical reaction of the liquid-liquid systems, and complex two-phase flow. Central composite design (CCD) was used to develop statistical models. These models were used to study the desired response and also determine the optimum combination of variables for optimizing purification of the tropical almond oil biodiesel ternary mixture system. The software used for experimental design was *MATLAB 8.5, 2015 version*. The dependent variables were a mass fraction of tropical almond oil biodiesel, glycerol, and methanol. Optimizing the operation of the central composite experimental design (CCD), a nine-level-two-factor central composite design was employed in carrying out the research resulting in the generation of thirty (30) experimental runs. The factors investigated in the research were temperature (°C) and withdrawal time intervals (mins). The extract phase components of the tropical almond oil biodiesel ternary system were output variables. The codes and the coding system designs are in Tables 1 and 2. The ranges of variables were based on preliminary studies conducted since the natural patterns connecting these dependencies were unknown. The obtained experimental data were analyzed using four different optimization models with the quadratic model giving the best output based on the assessments from the analysis of variance (ANOVA) for each model in terms of accuracy of prediction, R^2, adjusted R^2, probability value (*p-value*), root mean square estimate (*RMSE*), and sum of square estimates (*SSE*). The best model (quadratic) is presented in this study. The experimental data obtained were analyzed employing response surface methodology using a polynomial regression model. The format of the RSM model used was a quadratic model (second-order polynomial model). The model optimized the purification of the extract phase components of the tropical almond oil biodiesel ternary system. The format for the regression model is given in Equation (1) (quadratic with interaction model). *MATLAB 8.5, 2015 version* software generated Equation (1).

$$W_1 = a_0 + a_1X_1 + a_2X_2 + a_3X_1X_2 + a_4X_1^2 + a_5X_2^2 \quad (1)$$

Where W_i represents the tropical almond oil biodiesel ternary components (tropical almond oil biodiesel, glycerol, methanol), X_1 is the withdrawal time interval in minutes, X_2 is the temperature in °C and a_i are the regression coefficients or regressors.

The factors and Levels of the central composite design for the tropical almond oil biodiesel ternary system is presented in Table 1.

Table 1. Factors and levels of the CCD for tropical almond oil biodiesel ternary system

Factors	Levels								
	-4	-3	-2	-1	0	1	2	3	4
Temp (ºC)	20	25	30	35	40	45	50	55	60
Component withdrawal time (mins)	2	4	6	8	10	12	14	16	18

Table 2 shows the central composite design (CCD) for a two-factor-nine-level design using coding system.

Table 2. The central composite design (CCD) for two-factor-nine-level design using coding system

Runs	Temperature, X_1 (ºC)	Time, X_2 (mins)
1	-4	-4
2	-3	-4
3	-2	-4
4	-1	-3
5	0	-3
6	1	-3
7	2	-2
8	3	-2
9	4	-2
10	-4	-1
11	-3	-1
12	-2	-1
13	-1	0
14	0	0
15	1	0
16	2	1

17	3	1
18	4	1
19\	-4	2
20	-3	2
21	-2	2
22	-1	3
23	0	3
24	1	3
25	2	4
26	3	4
27	4	4
28	1	0
29	0	1
30	0	1

The independent and dependent variables were fitted to the chosen model equations. They were examined in terms of the goodness of fit. The goodness of fit of the regression equations was evaluated by the coefficient of determination (R^2), adjusted R^2; root mean square estimate (*RMSE*), sum of squares estimate (*SSE*), and the coefficient of relation (R). Analysis of variance (ANOVA) was employed for statistical testing of the models. The procedure was required to test the significance and adequacy of the models. The optimization of the process condition in terms of the purity of the biodiesel level in the ternary mixture of the extract phase (biodiesel phase) was carried out to obtain optimum purification of the biodiesel system. A graphical method was used to determine the optimum response and inputs.

3. Results and discussion

3.1 Response surface methodology (RSM) optimization/prediction for extract phase of the tropical almond oil biodiesel ternary system.

Second-order polynomial (quadratic interaction) model (Equation 1) was used to predict/ optimize the response and determine the output variables at which optimized outputs would be obtained. The regression coefficients are specific components of the ternary mixture.

Table 3 shows the central composite design for the extract phase of the tropical almond biodiesel system at the investigated conditions.

Table 3. Central composite design of two-factor-nine-level design using coding system for extract phase composition (Tropical almond oil biodiesel (w_1) + Methanol (w_2) + Glycerol (w_3) System)

Runs	Input variables (Factors)		Output variables		
	Temp (x_1)	Time (x_2)	w_1	w_2	w_3
1	20	2	0.940	0.040	0.020
2	25	2	0.930	0.053	0.017
3	30	2	0.920	0.065	0.015
4	35	4	0.920	0.070	0.010
5	40	4	0.920	0.070	0.010
6	45	4	0.928	0.063	0.009
7	50	6	0.920	0.070	0.010
8	55	6	0.910	0.080	0.010
9	60	6	0.900	0.090	0.010
10	20	8	0.950	0.040	0.010
11	25	8	0.950	0.040	0.010
12	30	8	0.950	0.040	0.010
13	35	10	0.923	0.068	0.009

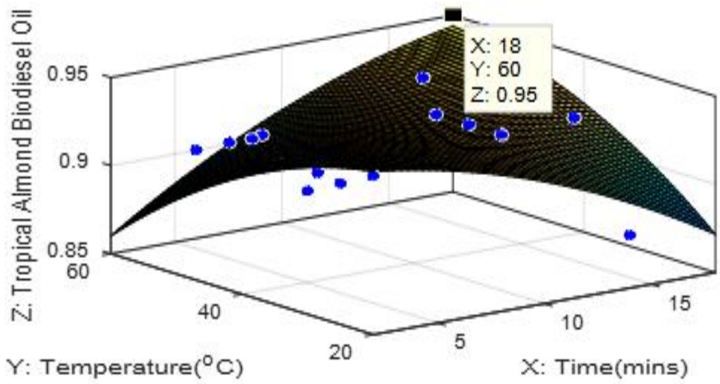
14	40	10	0.920	0.070	0.010
15	45	10	0.910	0.080	0.010
16	50	12	0.930	0.055	0.015
17	55	12	0.915	0.068	0.017
18	60	12	0.900	0.080	0.020
19	20	14	0.880	0.100	0.020
20	25	14	0.905	0.083	0.012
21	30	14	0.910	0.065	0.025
22	35	16	0.925	0.063	0.012
23	40	16	0.920	0.070	0.010
24	45	16	0.930	0.068	0.002
25	50	18	0.945	0.045	0.015
26	55	18	0.948	0.038	0.014
27	60	18	0.950	0.030	0.020
28	45	12	0.945	0.043	0.012
29	40	14	0.940	0.050	0.010
30	40	14	0.940	0.050	0.010

Analysis of variances (ANOVA) of RSM model prediction for extract phase is presented in Tables 4.

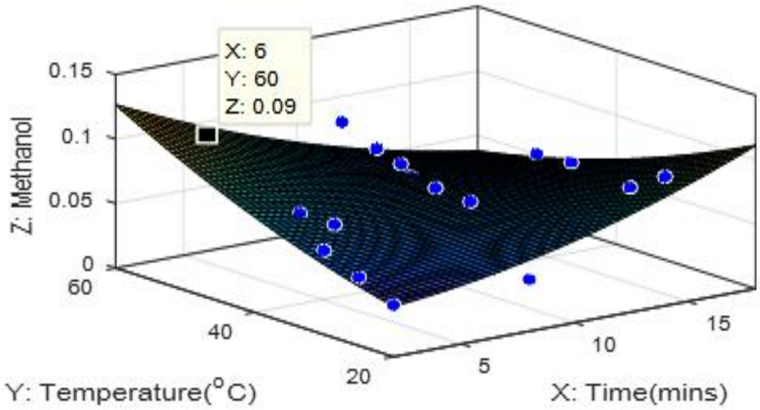
Table 4. ANOVA of RSM for tropical almond oil biodiesel ternary mixture system with quadratic regression model

Regressors	Tropical almond oil biodiesel (W_1)		Methanol (W_2)		Glycerol (W_3)	
	Regressor values	Statistical parameters	Regressor values	Statistical parameters	Regressor values	Statistical parameters
a_0	0.9502	R^2 = 0.9276	0.0088	R^2 = 0.9039	0.0407	R^2 = 0.9163
a_1	-0.0055	$AdjR^2$ = 0.9092	0.0060	$AdjR^2$ =	-0.0007	$AdjR^2$ =
a_2	0.0005	MSE =	0.0009	0.8914	-0.0014	0.9001
a_3	0.0002	0.00018054	-0.0003	MSE =	0.0001	MSE =
a_4	-0.0002	$RMSE$ = 0.0134	0.0002	0.00014338	0.0001	0.000018
a_5	-0.0000		0.0001	$RMSE$ = 0.0120	0.0001	$RMSE$ = 0.0042

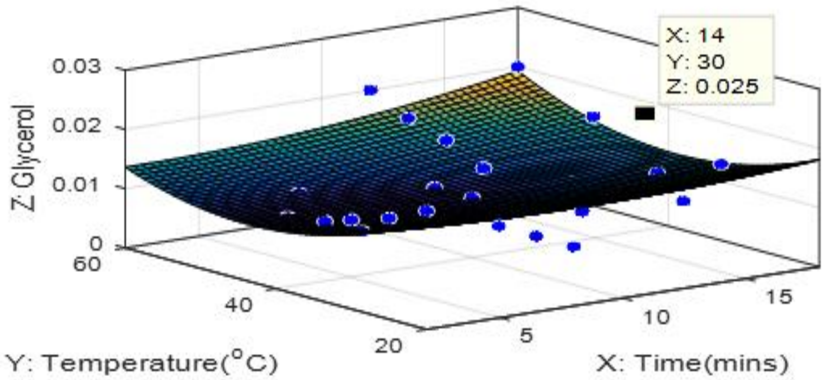
The prediction of mass fraction of withdrawn components at different temperatures and withdrawal times are presented as 3D plots in Figure 1 representing withdrawn mass fractions of tropical almond oil biodiesel, methanol and glycerol respectively. The 3D plots of each of the extract phase components (biodiesel, methanol and glycerol) are shown with each figure having a data tip indicating optimum (maximum) components withdrawn with respect to time and temperature. The extract phase mixture composition as shown in the 3D plots of Figure 1 reveals that optimal composition for tropical almond oil biodiesel was achieved at composition of 0.95 (95%), temperature of 60 °C and withdrawal time interval of 18 mins. The glycerol and methanol components composition of the mixture were also indicated at their respective temperatures and withdrawal times. The optimal compositions, withdrawal time interval and temperature of both methanol and glycerol in the extract phase were observed to be 0.09 (9%), 6 mins, 60 °C and 0.025 (2.5%), 14 mins, 30 °C respectively. The values of the optimal compositions revealed that low quantity of both methanol and glycerol were present at the optimized process condition giving a better indication for optimal purification at the observed process conditions for commercial viability. These values were in agreement with other studies conducted [8, 10, 12, 15].



A



B



C

Figure 1. 3D plots for extract phase mixture composition of withdrawn mass fractions of **(A)** tropical almond oil biodiesel, **(B)** methanol and **(C)** glycerol respectively

The model equation for tropical almond oil biodiesel optimization/prediction from extract phase is given as:

$$y = 0.91967 + 0.000111x_1 + 0.000344x_2 + 3.0539 \times 10^{-06}x_1x_2 - 2.6786 \times 10^{-06}x_1^2 + 4.8524 \times 10^{-05}x_2^2 \quad (2)$$

The model equation for the methanol optimization/prediction of extract phase is given as:

$$y = 0.06441 + 1.5853 \times 10^{-05}x_1 - 0.000151x_2 - 6.1348 \times 10^{-06}x_1x_2 + 1.7018 \times 10^{-06}x_1^2 - 4.1472 \times 10^{-05}x_2^2 \quad (3)$$

The model equation for glycerol optimization/prediction of extract phase is given as:

$$y = 0.013428 + 2.316 \times 10^{-07}x_1 - 0.000108x_2 + 1.9142 \times 10^{-06}x_1x_2 - 6.4665 \times 10^{-07}x_1^2 - 8.2769 \times 10^{-06}x_2^2 \quad (4)$$

The second-order polynomial regression model (Equation 1) was used to predict the response and determine the output variables at which optimized outputs would be obtained. This approach was also undertaken by several authors [1, 8-10, 11-13]. The regression coefficients are specific components of the ternary mixture. The model response was predicted using the regression coefficients for the specific components in order to obtain the best model for enhancing the extract (biodiesel) phase separation and purification of the three specific components in the mixture using response surface methodology. The final equations in terms of the coded factors for the central composite response surface quadratic equation model for each component of the phase namely; tropical almond oil biodiesel, methanol and glycerol ternary system are given in Equation (2) for tropical almond oil biodiesel optimization/prediction from the extract phase; Equation (3) for methanol optimization/prediction of the extract phase and Equation (4) for the glycerol optimization/prediction of the extract phase of the ternary mixture composition. The predicted values of the specific components (tropical almond oil biodiesel, methanol and glycerol) in the mixture obtained ranged from 0.880 mg/ml to 0.980 mg/ml for the tropical almond oil biodiesel; 0.030 mg/ml to 0.100 mg/ml for the methanol and 0.009 mg/ml to 0.025 mg/ml for the glycerol as seen in Table 3. The goodness of the quadratic model equations developed was checked using different criteria. The coefficient of determination (R^2) for model prediction of the tropical almond oil biodiesel component in the extract phase was 0.98126 which implied that 98.126 % of the component variation in the extract phase was attributed to the independent variable. The corresponding analysis of variance (*ANOVA*) is shown in Table 4. The *ANOVA* for the methanol and glycerol components are also shown in Table 4 respectively with their corresponding goodness of fit of the model equations based on the different criteria. The significant of each coefficient was determined by the *p*-values. The smaller the *p-values*, the more significant are the corresponding coefficient [22, 23]. The low *p-values* justify the significance of each term of the polynomial models developed. Each model has high values of R^2 and adjusted R^2 and these values show the suitability of the models by describing the extent to which responses are reflected [8, 9].

The *3D* response plots described by the regression model equations for the specific component of the extract phase were drawn to depict the influence of each variable (temperature and time) upon the response variable (specific components composition). This is shown in Figure 1. The quadratic model terms significantly affected the measured response of the system. Figure 1A shows the 3D plots for extract phase mixture composition of withdrawn mass fraction of tropical almond oil biodiesel system. It shows the effect of temperature and withdrawal time interval on the component composition of tropical almond oil biodiesel in the mixture. An optimal component composition of 0.95 mg/ml (95.0 %) of tropical almond oil biodiesel was obtained at a temperature of 60 °C and time of 18 mins. Figure 1B equally shows the influence of temperature and time on methanol component in the ternary mixture. The figure revealed that 0.09 mg/ml (9 %) methanol was obtained from the mixture at temperature of 60 °C and time of 6 mins and Figure 1C showed that the optimal glycerol component composition was achieved at 30 °C and time of 14 mins. This was 0.025 mg/ml (2.5 %). The result of this study revealed that the optimal component compositions for the ternary mixture of tropical almond oil biodiesel, methanol and glycerol in terms of phase separation and purification could easily be achieved at temperatures of 30 °C and 60 °C and time interval of between 14mins – 18 mins for all three components in the extract phase. This was in agreement with an earlier study conducted [19]. The optimal component values at these conditions were observed to be 95% (tropical almond oil biodiesel), 9% (methanol) and 2.5% (glycerol). The low values for methanol and glycerol are in agreement with ASTM D 975, ASTM D 6751 and EN 14214 standards for presence of methanol and glycerol in any biodiesel system [19, 20, 21]. The knowledge of these optimal conditions would help in determining the appropriate controller and sensors to be used in designing equipment for the phase separation and purification process [22, 23]. The results showed that optimal enhancement for phase separation and purification could be achieved at moderate factor values which are advantageous based on economics of biodiesel production.

4. Conclusion

Optimization of the extract phase in the purification of tropical almond oil biodiesel using response surface methodology (RSM) was carried out in this research. Second-order polynomial model was used to predict and optimize the response and determine the output variables at which the optimized outputs were obtained. The optimal components composition, temperature and withdrawal time were validated as 0.95 mg/ml (95%), 60 °C, 18 mins; 0.09 mg/ml (9%), 60 °C, 6 min; and 0.025 mg/ml (2.5%), 30 °C, 14 mins for tropical almond oil biodiesel, methanol and glycerol components respectively. The optimal performances of RSM optimization and prediction model was quite close and showed high levels of efficiency such that the following high values of coefficients of determination were obtained: 0.98126, 0.955845 and 0.98215 for tropical almond oil biodiesel, methanol and glycerol components respectively. The model equation for each of the optimized and predicted components was in agreement with experimental values obtained for the various components of the ternary mixture. This showed high degree of effectiveness for enhancing purification of tropical almond oil biodiesel system.

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