Optimization of Oil Extraction from *Thevetia Peruviana* **(Yellow Oleander) Seeds: A Case Study of Two Statistical Models**

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Abstract

In this study, optimization of oil extraction from Thevetia peruviana seeds was carried out using two statistical models which are Response Surface Methodology (RSM) and Artificial Neural Network (ANN). The physiochemical properties of the extracted seed oil were determined using standard methods. Results show that the optimal oil yield for both models (RSM and ANN) was established at sample weight of 40 g, solvent volume of 225 ml, and extraction time of 60 minutes. The theoretical oil yield predicted under this condition by RSM and ANN was 44.04 % and 43.95 % respectively, this was validated as 44.00 % (w/w). The physicochemical properties of the extracted oil showed the physical state of the oil to be liquid/orange-yellowish at room temperature, acid value of 1.8048 (mgKOH/g oil), Free Fatty Acid (FFA) of 0.9024 (%), iodine value of 97.60 (g I2/100g oil), saponification value of 57.5025 (mgKOH/g oil), and cetane number of 140.998 among others, these were compared with previous studies. The results showed that for Thevetia peruviana oil (TPO) extraction ANN was a better and more efficient optimization tool than RSM due to its higher value of R² (99.87 % and 99.936 % for RSM and ANN respectively).

Keywords: Thevetia peruviana oil, ANN, RSM

1.0 INTRODUCTION

Thevetia peruviana is a plant that is grown as hedges and kept for its bright and attractive flowers, and has been grown in Nigeria for over fifty years basically as an ornamental plant in homes, schools and churches by missionaries and explorers (Ana *et al.,* 2015). A yellow oleander tree can produce about 400–800 fruits all the year round depending on the climatic conditions and age of the plant. The fruits are usually green in colour and become black on ripening. Azam *et al*. (2005) reported that *Thevetia peruviana* seed has a high level of oil content (about 60-65%) in its kernel, and 40-45% valuable protein content in the seed, with major glyceride of palmitric, steric, and linoleic acid (Ibiyemi *et al*., 2002; Dhoot *et al*., 2011). However, it's nonedible due to the presence of cardiac glycoside (toxins) hence the plant has low significant economic value whereas it has a lot of potentials such biofuel production (Dhoot *et al*., 2011).

Oil extraction methods include solvent extraction, mechanical pressing, pressurized solvent extraction, soxhlet extraction, ultra-sonic extraction, Aqueous Enzymatic Oil Extraction (AEOE), stirring and shaking extraction method. Mechanical pressing is widely used but it produces oil with low value. Oseni *et al.* (2012) extracted oil from yellow oleander seeds by mechanical expression using a screw press as described by McCabe *et al*. (1987). Rodríguez *et al.* (2012) reported that using supercritical $CO₂$ extraction yielded more oil than that obtained from using solvent extraction. The oil produced has high purity, although it has high operating and investment costs when compared with solvent extraction. Solvent extraction has various advantages including high yield, less turbidity, and relatively low cost when compared to supercritical fluid extraction (Khraisha, 2002; Herodez *et al*., 2003; Jabar *et al.,* 2015). Kian *et al.* (2011) carried out solvent extraction of oil from *Jatropha curcus* while Kakati *et al*. (2014) extracted TPO with petroleum ether as solvent in a soxhlet extractor; Deka *et al.* (2011) extracted TPO with petroleum ether, hexane and diethyl ether to examine the extractability of the oil and it was reported that petroleum ether and hexane were equally good. Ana and Udofia (2015) extracted oil from *Thevetia Peruviana* seeds using soxhlet and cold-solvent extraction methods. Hexane-only (H-only) was used in the soxhlet while Hexane/Ether (H/E) mixture and H-only were respectively used in the cold extraction. Alabi *et al*. (2013) also extracted oil from *Thevetia peruviana* seeds using cold extraction method. Response Surface Methodology (RSM) employs multiple regression and correlation analyses as tools to assess the effects of two or more independent factors on the dependent variables (Jeong, *et al*., 2009). RSM has the advantage of reducing the number of experimental runs, which is sufficient to provide statistically acceptable results (Betiku *et al*., 2012). It has been used extensively in optimizing extraction of edible and non-edible oils from different oil sources such as pumpkin, palm oil, silkworm pupae, *Vetiveria zizanioides*, locust bean, rubber seed, to mention but a few (Danh *et al*., 2009; Mitra *et al*., 2009; Tan *et al*., 2009; Akinoso and Raji, 2011, Asuquo *et al.,* 2012, Bello and Otu 2015). ANN is a learning system based on a computational technique that can simulate the neurological processing ability of the human brain and can be applied to quantify a non-linear relationship between connecting factors and actual responses by means of iterative training of data obtained from a designed experiment (Bourquin *et al*., 1998a). ANN show superiority as a modelling technique for data sets showing non-linear relationships, and thus it is used for both data fitting and prediction abilities (Bourquin *et al*., 1998a, 1998b). It has been used to solve myriads of problems in the field of medicine, metrology, neurology, biology, psychology, science, mathematics and engineering (Sulaiman *et al*., 2010). Studies where both models have been used include; Sarve *et al*. (2015) and Betiku and Ajala (2014), where the former carried out response surface optimization and ANN modelling of biodiesel production from crude mahua (*Madhuca indica*) oil under supercritical ethanol conditions using $CO₂$ as co-solvent and the latter carried out modelling and optimization of TPO biodiesel synthesis via *Musa paradisiacal* (plantain) peels as heterogeneous base catalyst: a case study of ANN vs. RSM. The objective of this research was to investigate the optimum conditions in the extraction of oil from *Thevetia peruviana* seeds using two different models in carrying out the extraction process. A design of experiment combined with RSM analysis was applied using Minitab 15.5 to determine the simultaneous effects of various parameters on the oil yield and the interaction between parameters, a three-level–three-factor design was generated, the Box– Behnken design offered better advantages when compared with factorial design or central composite design by producing fewer number of experiments (Charoenchaitrakool and Thienmethangkoon 2011).

2.0 MATERIALS AND METHODS

2.1 Materials

The matured oilseeds of *Thevetia peruviana* used were collected from Government Secondary School, Ile Ife, Osun state, Nigeria. Seed coats were removed by hammer, before sun drying to obtain moisture free seeds, the seeds were oven dried at 60° C for 6 h before they are milled into powder by a blender. TPO was extracted using soxhlet extraction methods. The reagents used include Methanol, Sodium Hydroxide, Sulfuric acid, Phenolphthalein, Hydrochloric acid, 0.5N Alcoholic potassium hydroxide. All reagents used were of analytical grade supplied.

2.2 Methods

2.2.1 Oil extraction procedures

Four 500 ml soxhlet apparatus and n-hexane as extraction solvent were used for this study. A known weight of powdered seeds was placed in the thimble of the soxhlet apparatus, while a known volume of n-hexane was placed in a 500 ml round bottom flask fixed to the end of the apparatus and a condenser was tightly fixed at the other end of the extractor. The temperature was set to 60°C. The extracted oil-solvent mixture was collected, recovered and distilled to obtain solvent-free oil. The oil yield obtained was weighed to calculate the percentage yield.

2.2.2 Analysis Methods

The saponification value of TPO was determined using the Association of Official Analytical Chemists methods (AOAC, 2000). Standard ASTM D6751 methods were used to evaluate, acid value (ASTM D664), density (ASTM D1298), specific gravity (ASTM D4052), cetane number (ASTM D613), and FFA (ASTM D5555). Peroxide value was determined based on American Oil Chemists" Society (AOCS, 1980). Iodine value was estimated by applying Wijs method (AOAC, 1993 and Pocklington, 1990).

2.2.3 TPO extraction experimental design

Experiments were carried out according to Box-Behnken response surface design which involves 3 factors and require 3 levels, which generated 15 experimental runs. Experimental factors and their range levels used in this study are shown in Table 1. Minitab 15.5 statistical software package was used to randomly generate runs of experiment shown in Table 2.

Table 1: Coding of experimental factors and levels.

Table 2: TPO coded experimental design

2.2.4 Statistical Analysis by RSM

The response obtained was oil yield $(R_F, w/w\%)$, a second degree polynomial equation was employed as an empirical regression model for a better understanding of the correlations between the factors and response, as shown in Eqn. 1. (Montgomery, 2001; Koohikamali *et al.,* 2012)

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

Where R_F is the response factor (oil yield); τ_0 is the offset term; τ_i are linear coefficients; τ_{ii} and τ_{ii} are the interaction effect, and X_i and X_j are the independent variables. Since three factors are involved in this study, the mathematical relationship becomes Eqn. 2.

$$
R_F = \tau_0 + \tau_1 X_1 + \tau_2 X_2 + \tau_3 X_3 + \tau_{1,1} X_1^2 + \tau_{2,2} X_2^2 + \tau_{3,3} X_3^2 + \tau_{1,2} X_1 X_2 + \tau_{1,3} X_1 X_3 + \tau_{2,3} X_2 X_3
$$
\n(2)

2.2.5 Optimization studies by ANN

ANN software, Neural Power version 2.5 (CPC-X Software) was used in this study. The ANN was designed according to Betiku and Ajala (2014) which consist of an input layer with three neurons, an output layer with one neuron and a hidden layer. The optimal network topology was determined by using only one hidden layer while the number of neurons in this layer and the transfer function of hidden and output layers (Sigmoid, hyperbolic tangent function (Tanh), Gaussian, linear, threshold linear and bipolar linear) were determined iteratively by developing several networks. Each ANN was trained using a default stopping criteria of 100,000 iterations. Other conditions and parameters were chosen as the default values of the software.

2.2.6 Model comparison

The two models used in this study were compared using the coefficient of determination (R^2) ; this was used to identify the best model. R^2 is a measure of the amount of the reduction in the variability of the response by using the repressor variables in the model. The accuracy of the model was determined by evaluating R^2 value. In which R^2 must be close to 1.0 (Ebrahimpour *et al.*, 2008; Betiku and Ajala 2014). The R^2 was calculated according to Betiku and Ajala (2014) in Eq. 3

$$
R^{2} = 1 - \sum_{i=1}^{n} \left(\frac{(x_{i,cal} - x_{i,exp})^{2}}{(x_{avg,exp} - x_{i,exp})^{2}} \right)
$$
(3)

Where n is the number of experimental data, $x_{i,cal}$ is the calculated values, $x_{i,exp}$ is the experimental values and $x_{\text{avg,exp}}$ is the average experimental values.

3.0 RESULTS AND DISCUSSION

3.1 Quality characterization of TPO

The quality of TPO extracted was characterised in term of the physiochemical properties which were compared with previous studies (Table 3) to determine its suitability as a feedstock for various application. At room temperature, TPO obtained was liquid, orangeyellowish colour, having moisture content of 0.0131 which was lower than that reported by Yarkasuwa *et al*. (2013). Specific gravity of the extracted oil was lower than studies by Ibiyemi *et al*. (2002) and Kakati *et al*. (2014) while density of the oil was lower than previous studies by Usman *et al*. (2009), Deka *et al*. (2011), Yarkasuwa *et al*. (2013) and Ogunneye *et al*. (2014) as shown in Table 3.

The low FFA content of TPO obtained in this study was indicative of the good resistance of the oil to hydrolysis. Acid and FFA values are 1.8048 and 0.9024 respectively; which were higher than studies by Deka *et al*. (2011), Kakati *et al*. (2014) and Ogunneye *et al*. (2014) but lower than study by Yarkasuwa *et al*. (2013). Oxidative stability of the oil can be determined by its peroxide value; peroxide value obtained in this study was higher than studies by Ibiyemi *et al*. (2002) and Kakati *et al*. (2014). The peroxide value obtained in this extraction process is between 20 to 40 mg/g at which oil becomes unstable with offensive odour in storage (Jauro *et al*., 2011; Jabar *et al*., 2015).

According to Gunstone, (2004) iodine value is a measure of unsaturation of triglyceride oil, the value obtained in this study was higher than previous studies by Ibiyemi *et al*. (2002), Usman *et al*. (2009), Deka *et al*. (2011), Yarkasuwa *et al*. (2013), Kakati *et al*. (2014) and Ogunneye *et al*. (2014). The iodine value of the extracted oil is within the range of semi drying oil, this showed that it is a potential raw material in paint, lacquer, resin, polyol, grease, lubricating oil and diesel production (Eromosele *et al*., 1998; Jabar *et al*., 2015). Saponification value indicates the average molecular weight of triglycerides in the oil, the value found in this study was lower than previous studies by Ibiyemi *et al*. (2002), Usman *et al*. (2009), Yarkasuwa *et al*. (2013), Kakati *et al*. (2014) and Ogunneye *et al*. (2014). Cetane number is a measure of the fuel's ignition delay and combustion quality, the higher the cetane number the more suitable it can be used in biofuel production. Since the standard specification of cetane number for biodiesel is minimum of 40 (Meher *et al.*, 2006; Ramos *et al*., 2008). The cetane number shows that it had a high fuel potential. It can be deduced that trans esterification of the TPO could improve its fuel properties.

3.2 Optimization of TPO extraction

Table 4 shows the experimental conditions in coded factors and results obtained for the two models (RSM and ANN) considered in this study. From the Table, the optimum yield was 44.00% (w/w) at extraction time of 60 mins, solvent volume of 225 ml and sample weight of 40 g. The predicted responses for RSM and ANN at the optimum variable condition were 44.04% (w/w) and 43.95% (w/w) respectively. The minimum oil yield was obtained at extraction time of 40 mins, solvent volume of 225 ml and sample weight of 40 g, while predicted responses for this condition for the two models (RSM and ANN) are 21.04% (w/w) and 21.00% (w/w) respectively.

Based on the experimental results, a model equation was developed by Minitab 15.5 statistical software in order to predict the response (oil yield) as an expression of the independent variables (Eqn. 4).

$$
R_F (w/w %) = -145.0 - 0.027X_1 + 7.023X_3 + 0.05577X_1^2 - 0.01692X_3^2 - 0.11000X_1X_3
$$
\n
$$
(4)
$$

Where R_F is the oil yield, X_1 is catalyst amount, X_2 is the reaction time, and X_3 is the methanol/oil ratio.

Table 5 summerizes the test of significance for every regression coefficients and analysis of variance (ANOVA) generated by Minitab 15.5 software. In terms of the P-value, the significance of each coefficient was evaluated. From the table, P-values of the model terms were significant, i.e. $p < 0.05$. the linear term (X_1) , interaction effect (X_1X_3) and two quadratic terms $(X_1^2$, and X_3^2) were remarkably significant. However, the linear term (X_3) was not significant.

 $R^2 = 99.87\% R^2$ (adjusted) = 99.80% R² (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 model was evaluated by F and P values using Fischer's and null-hypothesis tests. The F-value predicts the quality of the entire model considering all design factors at a time. The P-value is the probability of the factors having very little or insignificant effect on the response. Larger F-value signifies better fit of the RSM model to the experimental data (Panwal *et al*. 2011). According to Datta and Kumar, (2012) F-value with low P-value indicates the high significance of the regression model. However, the Pvalue should be lower than 0.05 for the model to be statistically significant (Patel *et al*, 2011). Based on studies by Panwal *et al*, (2011), Patel *et al*, (2011), Datta and Kumar, (2012), the regression model found in this study was highly significant as denoted by the large F-value and low P-value of 1370.31 and 0.000 respectively. The "lack of fit" of low F-value and high P-value suggests that the model is not significant relative to the pure error; this was in conformance with study by Betiku and Ajala (2014). Non-significance of "lack of fit" was required for the model to fit.

To test the fit of the model equation, the regression model was determined by (determination coefficient) R^2 which provides a measure of how much variability in the observed response values can be explained by the experimental factors and their interactions (Sudamalla *et al.*, 2012). The R^2 value is always between 0 and 1 (Haider and Pakshirajan 2007; Liu and Wang 2007) however, for a good fit model it was recommended that R^2 should not be less than 80% (Joglekar and May, 1987). Results in Table 5 showed that R^2 value was 99.87% which signified 99.87% of variability in the observed response values could be explained by the model while only 0.13% of variability in the observed response values cannot be explained by the model. The remaining R^2 value of 0.13% of the total variations would be due to other factors which were not included in the model.

The adjusted R^2 was a corrected value for R^2 after the elimination of unnecessary model terms. The adjusted \mathbb{R}^2 was remarkably smaller than the \mathbb{R}^2 which implied that many nonsignificant terms were included in the model (Fang, *et al* 2010). In this study, it was found that the adjusted R^2 was smaller and very close to the R^2 and the respective values of adjusted R^2 and R^2 are 99.80% and 99.87%. The Predicted R^2 of 99.77% was in reasonable agreement with the Adjusted \mathbb{R}^2 of 99.80%. The adjusted \mathbb{R}^2 and \mathbb{R}^2 values indicate a high dependence and correlation between the observed and predicted responses. The accuracy of the prediction model obtained by regression analysis was verified by a linear correlation of predicted versus actual response values for TPO extraction by both models (Fig 1). The plots show that the models are adequate without any violation of independence or constant assumption.

Fig. 1: Linear correlation between experimental predicted response values for both models. The results of statistical analysis include the (regression coefficient) T and P values for linear, quadratic and combined effects of the variables given in Table 6. The significance of each coefficient in the experimental model was determined by T and P- value using Minitab 15.5. A high T-test value and a low P-value indicated by the model showed that the linear coefficient (X_1) and the quadratic term (X_1^2) have significant effects $(P > |T| < 0.05)$ on the oil yield (Niladevi *et al*., 2009; Baoxin *et al*., 2011). All other terms displayed on table are not significant. Using the experimental results, low values of standard error was observed in the intercept and all the model terms, these indicate that the regression model fits the data, and the prediction was good. The F ratio was calculated for 95% level of confidence interval (CI) and variance inflation factor (VIF) obtained in this study showed that the centre points were orthogonal to all other factors in the model (Table 6).

Table 6: Regression coefficients and significance of response surface quadratic

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Graphical depiction are used to illustrate the relationship between the response and experimental levels of each variable and the type of interactions between test variables to deduce the optimum conditions, this was illustrated by varying two variables while holding the other variables constant (Datta, and Kumar, 2012), this was demonstrated in Fig $2 - 7$ as 2D contour and 3D surface plots. The lowest oil yield (21%) was observed at the smallest sample weight of 40 g and lowest extraction time of 40 mins; but at equal solvent volume of (225 ml), increased sample weight (50 g) and extraction time (60 mins) yielded more oil (33%). The combination of highest sample weight and highest extraction time significantly increase the oil yield by 12%. However, for both models (RSM and ANN) the optimal condition for oil yield was established at sample weight of 40 g, solvent volume of 225 ml and extraction time of 60 mins. The theoretical oil yield predicted under this condition by both models (RSM and ANN) are 44.04% and 43.95% respectively, these was validated as 44.00% using three experimental replicates under the optimal condition, which confirmed the efficacy of the models. The importance of effective parameters on percentage oil yield by ANN was illustrated in Fig 8, this shows that the significance of sample weight was higher than extraction time while solvent volume has no significance at all.

Fig. 2: RSM contour and surface plots showing interactive effect of extraction time, solvent volume on oil yield keeping sample weight constant.

Fig. 3: RSM contour and surface plots showing interactive effect of sample weight, extraction time on oil yield keeping solvent volume constant.

Fig. 4: RSM contour and surface plots showing interactive effect of sample weight, solvent volume on oil yield keeping extraction time constant.

Fig. 5: ANN three dimensional plot showing effect of solvent volume, extraction time and their mutual effect on oil yield.

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their mutual effect on oil yield.

Fig. 7: ANN three dimensional plot showing effect of sample weight, solvent volume and their mutual effect on oil yield.

Fig. 8: Importance of effective parameters on percentage oil yield.

3.3 Model comparison

The accuracy of both models (RSM and ANN) were determined by comparing the value of $R²$. The result depicts that both optimization tools gave good predictions due to the value of R² (99.870% and 99.936% for RSM and ANN respectively). Therefore, for TPO extraction ANN showed a clear superiority over RSM because of the higher value of \mathbb{R}^2 .

4.0 Conclusion

Thevetia peruviana seeds were found to be rich in oil, which should be considered as a suitable economical feed stock. The optimal condition of the variables was established at extraction time of 60 mins, solvent volume of 225 ml and sample weight of 40 g, using these optimal factor values under experimental conditions in three independent replicates, an average content of 44.00% (w/w) was achieved. The quality of oil extracted from *Thevetia peruviana* oilseed under optimal condition can serve as feedstock for many industrial applications such as in soap industries, paint industry, making of grease and lubricants, in feedstock formulation and production of biodiesel.

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