Journal of Oleo Science
Copyright ©2024 by Japan Oil Chemists' Society
doi : 10.5650/jos.ess24022
J. Oleo Sci. **73**, *(9) 1149-1158 (2024)*

JOURNAL OF OLEO SCIENCE JOS

Investigation of Factors Affecting Extraction of *Calophyllum inophyllum* Seed Oil via Response Surface Methodology

Eriola Betiku[1]*, Esther A. Olatundun[2], Damilola A. Taiwo[2], Olumayowa F. Omotunde[2], Victor I. Omofaye[2], Biola A. Babalola[2], Adebisi A. Agboola[2], and Lekan M. Latinwo[1]

[1] *Department of Biology, Florida Agricultural and Mechanical University, Tallahassee, FL 32307, USA*
[2] *Department of Chemical Engineering, Obafemi Awolowo University, Ile-Ife 220005, Osun State, NIGERIA*

Abstract: This study used the Soxhlet apparatus to investigate honne oil (HO) extraction optimization. Twenty-four (24) experiments were formulated using the D-optimal design considering extraction time (2 - 6 h), honne weight (20 - 60 g), and particle size using acetone. The yield, functional groups, physical and chemical properties, and fatty acid composition of the HO were assessed. The optimal extraction conditions established were a time of 6 h, fine particle size, and honne weight of 20 g with a high HO yield of 70.85 wt.%. The HO had an acid value and kinematic viscosity of 35.68 mg KOH/g oil and 52.96 mm^2/s, respectively. The observed coefficient of determination of 0.9870 suggests that the model developed for the process is efficient. The functional groups and fatty acids of the HO confirm that it is highly unsaturated with the regions of trans-unsaturation bending vibrations and double bond stretching. The properties of the HO demonstrate that it could be used to produce biodiesel, notwithstanding the necessity for pretreatment.

Key words: *Calophyllum inophyllum*, modeling, optimization, D-optimal, oil extraction

1 Introduction

The ability of a specific oilseed to be valuable in the industries relies upon its production rate, potential application, accessibility, and simplicity of the processing technology. Accordingly, while a significant portion of oilseeds are underutilized, the oil processing industry uses some oilseeds in significant amounts regularly. Since it is becoming gradually clear that first-generation biofuels, initially made from food crops and primarily oilseeds, cannot achieve goals for biofuel generation, environmental change mitigation, and economic development, sustainable production of renewable energy is receiving serious consideration on a global scale[1]. Non-edible oils are in abundance and quite cost-effective compared to edible oils. Using non-edible oils instead of edible ones can lower production costs and avoid food security concerns[2]. Some examples of these non-edible oils are soapnut (*Sapindus mukorossi*)[3], yellow oleander (*Thevetia peruviana*)[4], Napoleon's plume (*Bauhinia monandra*)[5], neem (*Azadirachta indica*)[6], honne (*Calophyllum inophyllum*)[7], kariya (*Hildergadia bateri*)[8], sandbox (*Hura crepitans*)[9].

Honne, a member of the Clusiaceae, is a mangrove species that originates mainly in the tropical and subtropical regions[10]. Honne is a source of bioactive materials[11], biolubricant[12], bio-dielectric fluid[13], biohydrogen and biochar[14], reducing sugar production[15], biosurfactant and biopolymer[16], bio-oil[17], and bioabsorbent[18]. It has been reported as the second-highest raw material for biodiesel production[10]. Some of the work reported in the literature on honne oil (HO) biodiesel production include conversion of HO to biodiesel using a catalyst produced from cocoa and plantain wastes[7], HO and lipase as the catalyst[19], and HO and NaOH as the catalyst[20], simultaneous esterification and transesterification HO with sulfuric acid[21].

There are various ways of extracting oil from oilseed. The most widely used methods are mechanical and solvent extraction. For commercial extraction and biodiesel production, oils are usually extracted from oilseeds using solvent extraction methods[22]. This method involves the solid-liquid extraction method, where oil pigments in dry solid with low moisture content are extracted into the solvent without changing the characteristics of the extracted oil pigments. The oil solubility in a solvent varies, which means that the properties of the solvent should be known

*Correspondence to: Eriola Betiku, Department of Biology, Florida Agricultural and Mechanical University, Tallahassee, FL 32307, USA
E-mail: eriola.betiku@famu.edu ORCID ID: https://orcid.org/0000-0003-4521-1277
Accepted June 17, 2024 (received for review January 29, 2024)
Journal of Oleo Science ISSN 1345-8957 print / ISSN 1347-3352 online
https://www.jstage.jst.go.jp/browse/jos/ https://mc.manuscriptcentral.com/jjocs

prior to the extraction process, as this could affect the oil yield owing to their polarity[8]. Solvent extraction has various influencing parameters, including time extraction, solvent type, temperature, and particle size[8, 22].

Optimization is an important method for the extraction process. It gives detailed information about the process. Response surface methodology (RSM) is a tool utilized to design and optimize responses and can be adapted for oil extraction processes. It has been applied during the extraction of oil from various oilseeds such as seeds from Napoleon's plume[5] and yellow oleander[23], sandbox[24], *M. oleifera*[25], rubber[26], kariya[8], shea butter[27]. D-optimal design is a statistical tool that is now in use with RSM to design an experiment. It is ideal for prediction performance since it can lower the dispersion of regression coefficients and generate highly uniform fractional design space curves for most of the design space[28]. It is different from central composite design and Box-Behnken design due to its ability to accept both quantitative and qualitative factors (that is, numeric and categoric factors)[8, 24, 25]. The method has been applied in oil extraction processes involving yellow oleander, rubber, *M. oleifera*, sandbox, and kariya seeds[8, 24–26].

In the present study, the HO extraction process was modeled and optimized by combining D-optimal design and RSM using Soxhlet apparatus and acetone, a renewable solvent. The D-optimal design was used to analyze the individual influence and interactions of the variables considered: quantitative factors (honne weight and time) and a qualitative factor (particle size). The properties of the oil extracted, *viz*. oil yield, functional groups, fatty acid profiles, physicochemical properties were determined for quality characterization and to ascertain its potential for biodiesel and oleochemical production.

2 Experimental

2.1 Materials

For this study, freshly matured honne fruits were collected from Ile-Ife town in Osun State, Nigeria. The chemicals and the reagents used are of analytical grade.

2.2 Honne seed preparation

The harvested honne fruits were sun-dried for two weeks, shelled, and decorticated to remove the kernels. These kernels underwent further 5-day sun drying to attain constant weight and reduce the moisture content. The dried kernels were pulverized with the aid of a manual grinding machine and coffee grinder to get the fine particle size, some were cracked into pieces to obtain the coarse particle size, and finally, some seeds were left whole to get the pebble particle size (Fig. 1).

2.3 HO extraction

In this study, solvent (acetone) with a Soxhlet apparatus was used to extract oil from honne kernels. The efficiency of the process was evaluated by different sizes of the honne kernels (fine (35 μm), coarse (0.47 cm), and pebble (1.34 cm)). A round bottom flask was connected to a Quickfit Soxhlet apparatus attached to a condenser. The acetone (200 mL) was poured into the flask while the honne sample (fine, coarse, and pebble) was placed in a muslin fabric in the extraction chamber. The temperature (60℃) used was a little above the boiling point of acetone (56℃). The extraction was followed by the distillation procedure for recovering the acetone from the oil-acetone mixture[8, 26]. The HO yield was established consistent with Equation (1).

$$\text{HO yield (wt:\%)} = \frac{\textit{weight of HO extracted (g)}}{\textit{initial weight of Honne seed kernel (g)}} \times 100 \quad (1)$$

2.4 Experimental design for the study

The experimental design approach employed in this work was a D-optimal design, which accommodates both numeric and categoric factors. Three factors were considered during this study: two numeric factors (honne weight and extraction time) and one categoric factor (particle size). Table 1 shows the 24 experiments that were generated, indicating required points (12), lack-of-fit points (6), and replicate points (6). The mean HO yield was reported after these laboratory experiments were conducted in duplicates.

Fig. 1 Processed seeds. (a) fine, (b) coarse, and (c) pebble.

Table 1 D-optimal design, actual and predicted HO yields.

No	Honne weight (g) X	Extraction Time (h) Y	Particle Size Z	Actual HO yield (wt.%)	Predicted HO yield (wt.%)
1	20	6	Pebble	7.43	3.6
2	20	4	Pebble	4.28	4.45
3	20	2	Pebble	2.87	4.62
4	20	2	Pebble	2.95	4.62
5	40	4	Pebble	3.93	4.35
6	60	2	Pebble	2.06	0.25
7	60	2	Pebble	2.19	0.25
8	60	6	Pebble	4.845	6.6
9	60	6	Pebble	4.83	6.6
10	20	4	Coarse	22.48	23.12
11	20	4	Coarse	22.80	23.12
12	40	2	Coarse	18.89	19.55
13	40	4	Coarse	27.28	23.54
14	40	6	Coarse	26.19	26.85
15	40	6	Coarse	26.35	26.85
16	60	2	Coarse	17.15	17.64
17	60	4	Coarse	23.01	23.47
18	20	2	Fine	60.58	55.46
19	20	4	Fine	61.73	63.44
20	20	6	Fine	68.10	70.75
21	40	2	Fine	35.42	36.91
22	60	2	Fine	15.13	17.88
23	60	6	Fine	45.35	40.53
24	60	6	Fine	39.21	40.53

2.5 Statistical analysis and model validation

The data obtained were statistically examined using multiple regression analysis and RSM. The data were fitted to the quadratic mathematical model equation. The significance of each factor investigated and their interactions were determined using analysis of variance (ANOVA). The model quality developed was determined using fit statistics, including coefficient of determination (R^2), adjusted R^2, predicted R^2, and adequate precision. The model was further evaluated using diagnostic plots such as parity, normal probability, and internally studentized residual plots. The model was validated using the optimal conditions obtained to conduct three replicate experiments in the laboratory[9)].

2.6 Characterization of HO

The physical and chemical properties of the HO obtained were determined, including acid, peroxide, iodine, and saponification values, and refractive index, kinematic viscosity, and density by methods previously described[29)]. Visual inspection was used to determine the physical condition and color of the HO sample, and they were then contrasted with reference colors under natural lighting. As previously presented in our earlier study, functional groups and fatty acid content of the HO were identified through Fourier transform infrared (FTIR) and gas chromatography techniques, respectively[8)].

3 Results and Discussion

3.1 HO extraction process modeling

The results of the HO extraction are displayed in Table 1. The HO yield observed ranged from 2.06 to 68.10 wt.%. The quadratic regression models representing the HO extraction process in terms of the actual factors for the fine, coarse, and pebble samples are depicted by Equations (2)-(4).

Table 2 Fit statistics and ANOVA results terms.

Source	Sum of squares	Degree of freedom	Mean value	F-value	p-value
X	538.97	1	538.97	53.5	0.0001
Y	425.94	1	425.94	42.28	0.0001
Z	6923.04	2	3461.52	343.61	0.0001
XY	39.24	1	39.24	3.9	0.0719
XZ	1059.06	2	529.53	52.56	0.0001
YZ	203.78	2	101.89	10.11	0.0027
X^2	0.19	1	0.19	0.019	0.8923
Y^2	0.43	1	0.43	0.043	0.8395
Model	9188.53	11	835.32	82.92	< 0.0001
ANOVA					
Residual	120.89	12	10.07		
Pure Error	18.93	6	3.15		
Corrected Total	9309.42	23			
R^2	0.9870				

Particle size = Fine

$$W = 66.7188 - 0.9832X + 3.5732Y + 0.0460XY - 0.0006X^2 - 0.0842Y^2 \quad (2)$$

Particle size = Coarse

$$W = 20.9379 - 0.1269X + 0.6562Y + 0.0460XY - 0.0006X^2 - 0.0842Y^2 \quad (3)$$

Particle size = Pebble

$$W = 7.4271 - 0.1529X + 0.5030Y + 0.0460XY - 0.0006X^2 - 0.0842Y^2 \quad (4)$$

The final model equation for the process in terms of coded factors is described by Equation (5).

$$W = 24.87 - 5.71X + 4.82Y + 21.86Z[1] - 1.34Z[2] + 1.84XY - 11.24XZ[1] + 5.88XZ[2] + 4.66YZ[1] - 1.17YZ[2] - 0.24X^2 - 0.34Y^2 \quad (5)$$

Where W = HO yield, X = honne weight, Y = extraction time, and Z = particle size, while [1] and [2] are the levels of each factor.

The summary of the overall quality of the model developed for the process (Equation (5)) is displayed in Table 2. The model terms were tested at a 95% confidence level. In this case, the linear terms (honne weight, time, and particle size), cross-products of honne weight and particle size (XZ), and time and particle size (YZ) are all significant, implying they had a great influence on the HO yield. The F-value of 82.92 and $p < 0.0001$ obtained from the ANOVA evaluation of the regression model support its significance, demonstrating that it is adequate for describing the extraction process.

The R^2 value signifies that the model can account for 98.70% of the predicted and experimental HO yields variance. It has been established that R^2 of ~1.0 indicates the model is appropriate[30], and the value should not be less than 0.80[31]. The predicted R^2 (0.9279) and adjusted R^2 (0.9751) for the model show that it is acceptable since their difference is < 0.2. The coefficient of variance of 13.98% implies a high correlation of the experimental data with the predicted data. A signal-to-noise ratio of 31.410 indicates a good signal for the model since it is > 4. Analytical plots were used to assess further the effectiveness of the established model (Figs. 2a-2d). Figure 2a represents the correspondence graph of predicted and experimental data. The fitted points nearly approach the reference line, illustrating a high correspondence.

Figure 2b shows the normal probability and internally studentized residual plot. The normal probability graph shows if the residuals are distributed normally or not. Since the points all fairly follow a straight line, it confirms the normality of the data[32]. Figure 2c illustrates the graph of studentized residuals and predicted HO yields. The random scattering of the residual indicates the adequacy of the regression model in describing the HO extraction process without violating the equality of variance[33]. The plot of residuals versus experimental run number requires that most standardized residuals (di) fall between the limits of −3 and 3[24, 25]. No data exceeded the range in Fig. 2d, indicating no outlier[33].

Figures 3a-3c show the response surface graphs of the sample weight and extraction time on HO yield with different particle sizes (fine, coarse, and pebble). The plots show the synergistic effect of time and honne weight on the HO yield. For all particle sizes, the time and honne weight are inversely proportional on the HO yield. The HO yield increases as honne weight reduces and extraction time increases. Based on the particle size, the HO yield trend

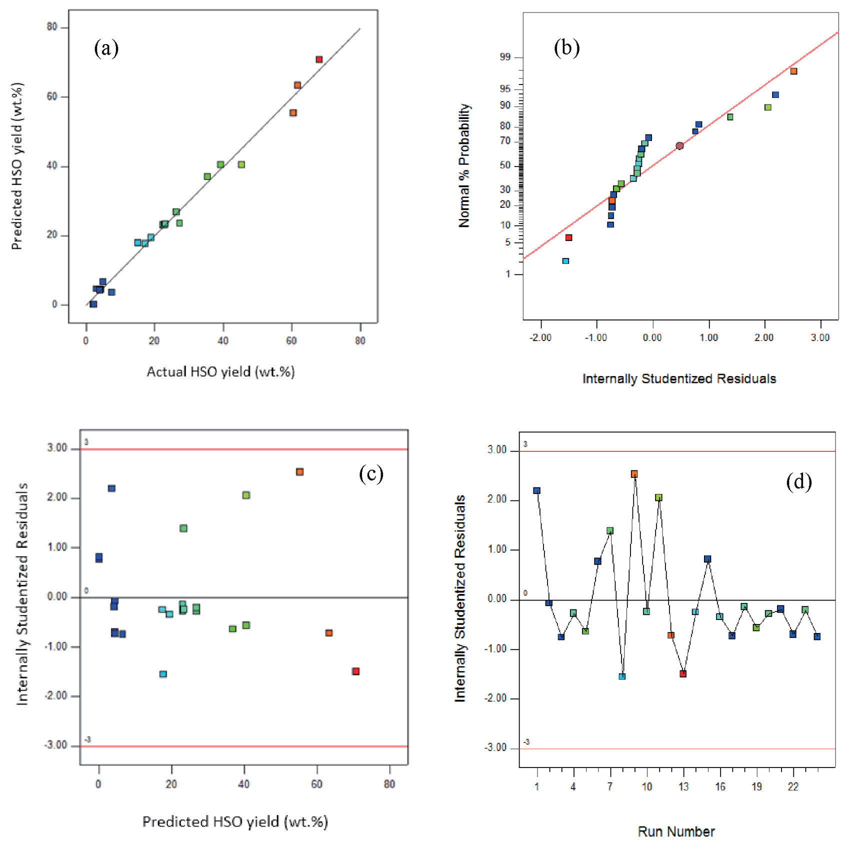

Fig. 2 Diagnostic plots for the HO extraction process model.

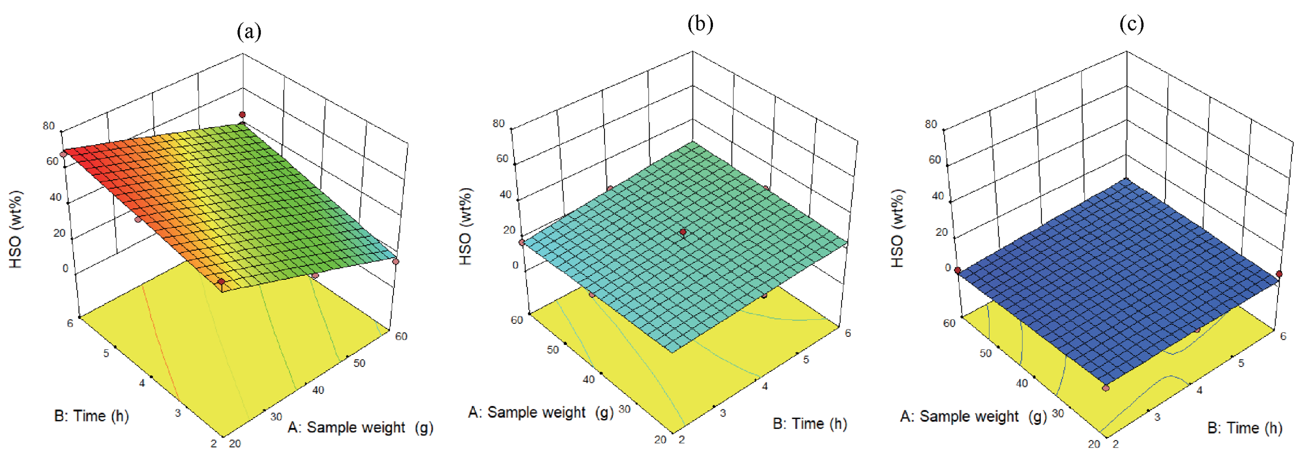

Fig. 3 The response surface plots of the HO extraction. (a) fine particle size. (b) coarse particle size. (c) pebble particle size.

follows the trend of fine > coarse > pebbles. The highest HO yield using fine particle size was 68.10 wt.% at a time of 6 h and honne weight of 20 g, while with coarse and pebbles, the highest yield was 27.28 and 7.43 wt.%, respectively. A similar observation on honne was reported using *n*-hexane[34]. The increase in the HO yield using fine particles is because of the increased surface area, allowing for the penetration of the solvent and boosting its diffusivity[35]. Comparable results on increased oil yield have been obtained for pulverized plant seed oil extraction from Kariya[8], rubber seed[26], *Hura crepitans*[9, 24], beniseed[36], yellow oleander seed[23], and moringa seed[25].

The extraction process variables were optimized to maximize HO yield. The predicted conditions were time of 6 h, honne weight of 20 g, and fine particle size with an estimated HO yield of 70.21 wt.%. The conditions were used

Table 3 Physicochemical properties of extracted HO.

Property	This study	Atabani *et al.*[22]	Belagur *et al.*[41]	Bhuiya *et al.*[34]
Physical state/color	Greenish yellow	–	–	–
Kinematic viscosity (mm^2/s) at 40℃	52.96	55.677	32.48	52.67[b], 53.18[c]
Density (g/cm^3) at 15℃	0.951[a]	0.951	0.910	0.937[b], 0.938[c]
Refractive index at 25℃	1.4780	1.4784	–	–
Cetane number	53.23	–	51 – 56	52.2[b], 52[c]
Acid value (mg KOH/g oil)	35.68	41.74	44.76	38.86[b], 39.22[c]
Calorific value (MJ/kg)	40.92	38.51	39.11	39.52[b], 38.10[c]
Iodine value (g I_2/100 g oil)	94.69	–	82 – 98	85[b], 85.3[c]
Saponification value (mg KOH/g oil)	186.53	–	91 – 202	192[b], 192.3[c]

a – measurement determined at 25℃; b – *n*-hexane; c – screw press.

Fig. 4 Samples of a) HO and b) HO biodiesel.

to conduct three replicated experiments, and the mean HO yield observed was 70.85 wt.%, validating the adequacy of the proposed model. When hexane was used for HO extraction with a Soxhlet apparatus, the yield reported was 46.22%[37]. Bhuiya *et al.*[34] observed an HO yield of 72.1% and 78% from whole seeds and grated seeds, respectively, using a screw press, while 86.4% was reported for extraction with *n*-hexane.

3.2 Quality of extracted HO

The HO quality in terms of its physical and chemical properties is described in Table 3. The HO had a dark greenish-yellow coloration (Fig. 4) and a 0.013% moisture content. The HO had a very high kinematic viscosity (52.96 mm^2/s), possibly due to its chemical structure and high molecular mass, indicating good lubrication properties[38]. The iodine value of HO (94.69 gI_2/100 g oil) suggests a high degree of unsaturation, resulting in glyceride polymerization. The iodine value measures the total unsaturation level of fatty acid mixtures[24, 39]. The more unsaturated bonds in an oil, the higher the iodine value. Oils with high mono-unsaturation levels are suitable feedstock for biodiesel production. The HO had an acid value of 35.68 mg KOH/g oil, implying a high free fatty acids (FFA) content (17.84%). A high FFA reduces the oxidative stability of oil, leading to soap formation during transesterification with low molecular alcohol. Hence, there is a need for the pretreatment of the HO before transesterification to obtain biodiesel[7]. The rancidity rate is associated with peroxide value, which varies according to the degree of unsaturation[40]. The HO is less prone to rancidity at room temperature since it is <10 meqO_2/kg oil and has a good shelf-life[9].

The HO properties are comparable to the values reported earlier by other workers (Table 3). Similarly, the properties of the HO compared well with other vegetable oils (Table 4). The HO has properties similar to those reported for the common feedstock used for biodiesel production, proving that the oil could serve as biodiesel feedstock. This assertion has been confirmed in our earlier work using the HO to produce biodiesel, as displayed in Fig. 4b[7].

Table 5 shows the fatty acid compositions of the HO compared with other sources. The fraction of unsaturated fatty acids is more dominant in the HO, with oleic acid as the highest (41.63 wt.%) followed by linoleic acid (30.71 wt.%). The result obtained is similar to those reported earlier in the literature, irrespective of the extraction method solvent, except for Rajendran *et al.*[47] with linoleic acid as the most dominant fatty acid.

The functional groups identified in the HO are depicted in Fig. 5. The bands at 3007.12 and 3011 cm^{-1} indicate the presence of unsaturated fatty acids, such as oleic and linoleic acids, resulting from the cis = CH stretch bands[48]. The asymmetric stretching vibration of =C-H in CH_2 shows a strong peak of 2926.11 cm^{-1} and symmetric stretching vibration at 2854.74 cm^{-1} for HO extracted with acetone[8, 47, 49].

The prominent peak at 1743.71 cm^{-1} for HO demonstrates C = O (ester) stretching vibration of the carbonyl group presence[8, 47]. Band peaks ranging from 1500-1200 cm^{-1} are the fingerprint region. The peaks at 1465.95, 1377.22, 1244.13, and 1163 cm^{-1} are assigned to –C–H

Table 4 Properties of HO oil compared with other vegetable oils.

Oil	Specific gravity	Kinematic viscosity (mm^2/s) at 40℃	Cetane number	Acid value (mg KOH/g)	Calorific value (MJ/kg)	Reference
Honne	0.951	52.96	53.23	35.68	40.92	This study
Rubber	0.91	66.2	37	34	37.5	42)
Mahua	0.96	24.58	–	38	36	43)
Palm	0.92	38.1	–	18.5	39.4	44)
Soyabean	–	32.6	37.9	–	39.623	45)
Neem	0.94	20.5 – 48.2	51	–	36.6	41)
Jatropha	0.92	18.2	33.7 – 51	3.8	38.5	46)

Table 5 Comparison of fatty acid compositions (wt.%) of HO from different sources.

Fatty acid	Structure	This study	Mofijur, *et al.*[50]	Sahoo, *et al.*[51]	Ramaraju and Kumar[52]	Silitonga, *et al.*[53]	Fattah, *et al.*[54]	Bhuiya *et al.*[34]
Palmitic	C16:0	14.7	14.7	12.01	17.9	14.2	13.9	13.6
Palmitoleic	C16:1	0.2	0.3	–	2.5	–	0.2	0.2
Stearic	C18:0	9.9	13.2	12.95	8.5	15.9	15.1	16.7
Oleic	C18:1	41.6	46.1	34.09	42.7	38.9	40.3	40.1
Linoleic	C18:2	30.7	24.7	38.26	13.7	28.1	25.6	26.3
Linolenic	C18:3	0.2	0.2	0.3	2.1	0.76	0.2	0.3
Others		2.6	0.8	2.39	12.6	2.14	4.7	1.2

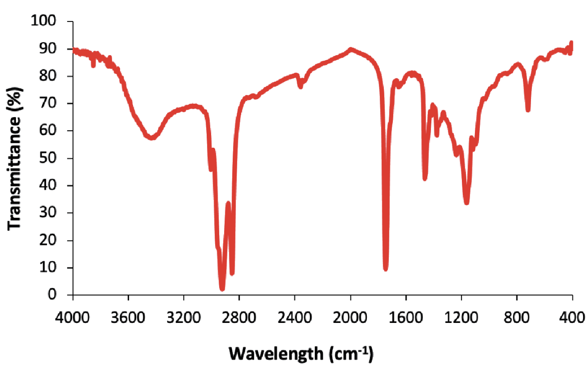

Fig. 5 Identification of functional groups in HO.

bending, –C–H (CH_3) bending (symmetric), –C–O, $-CH_2-$ (stretching, twisting out of plane vibration) and strong –C–O, $-CH_2-$ (stretching vibration, bending), respectively[8, 24, 25].

4 Conclusions

HO was successfully extracted from honne seeds by the Soxhlet apparatus using acetone. In this work, an investigation of the impact of sample size, honne weight, and time on the HO yield was performed. The results revealed that a reduction in particle size boosts the extraction rate, which results in a high HO yield. The best model predicted extraction conditions for the process are a time of 6 h, honne weight of 20 g, and fine particle size with a HO yield of 70.21 wt.%. The model validation was conducted in replicated experiments using the optimum values, and an average HO yield of 70.85 wt.% was observed. The physicochemical properties, fatty acid profile, and functional groups of the HO demonstrated that the oil could serve as a cheap source of biodiesel feedstock and, since it is non-edible, will not compete with food. The results of this study would help design the best conditions for maximizing the oil extraction from honne seeds.

Acknowledgements

EB acknowledges the provision of workspace by Department of Biological Sciences, FAMU.

References

1) Ahmad, A.L.; Yasin, N.M.; Derek, C.J.C.; Lim, J.K. Microalgae as a sustainable energy source for biodiesel production: a review. *Renew. Sustain. Energy Rev.* **15**, 584-593 (2011).
2) Das, P.; Jha, C.K.; Saxena, S.; Ghosh, R.K. Can biofuels help achieve sustainable development goals in India?

A systematic review. *Renew. Sustain. Energy Rev.* 192, 114246 (2024).
3) Panda, A.; Kumar, A.; Mishra, S.; Mohapatra, S. Soapnut: A replacement of synthetic surfactant for cosmetic and biomedical applications. *Sustain. Chem. Pharm.* 17, 100297 (2020).
4) Betiku, E.; Ajala, S.O. Modeling and optimization of *Thevetia peruviana* (yellow oleander) oil biodiesel synthesis via *Musa paradisiaca* (plantain) peels as heterogeneous base catalyst: A case of artificial neural network vs. response surface methodology. *Ind. Crops Prod.* 53, 314-322 (2014). doi: 10.1016/j.indcrop.2013.12.046
5) Akintunde, A.M.; Ajala, S.O.; Betiku, E. Optimization of Bauhinia monandra seed oil extraction via artificial neural network and response surface methodology: A potential biofuel candidate. *Ind. Crops Prod.* 67, 387-394 (2015).
6) Agu, C.M.; Ani, K.A.; Ani, O.N.; Nnaji, P.C.; Kadurumba, C.H.; Esonye, C. Application of efficient soft computing approaches for modeling methyl ester yield from *Azadirachta indica* (Neem) seed oil: A comparative study of RSM, ANN and ANFIS. *Green Technologies and Sustainability* 2, 100057 (2024).
7) Olatundun, E.A.; Borokini, O.O.; Betiku, E. Cocoa pod husk-plantain peel blend as a novel green heterogeneous catalyst for renewable and sustainable honne oil biodiesel synthesis: A case of biowastes-to-wealth. *Renew. Energy* 166, 163-175 (2020).
8) Okeleye, A.A.; Betiku, E. Kariya (*Hildegardia barteri*) seed oil extraction: comparative evaluation of solvents, modeling, and optimization techniques. *Chem. Eng. Commun.* 206, 1181-1198 (2019).
9) Omilakin, R.O.; Ibrahim, A.P.; Sotunde, B.; Betiku, E. Process modeling of solvent extraction of oil from *Hura crepitans* seeds: Adaptive neuro-fuzzy inference system versus response surface methodology. *Biomass Convers. Biorefin.* 13, 247-260 (2023).
10) Jain, M.; Chandrakant, U.; Orsat, V.; Raghavan, V. A review on assessment of biodiesel production methodologies from *Calophyllum inophyllum* seed oil. *Ind. Crops Prod.* 114, 28-44 (2018).
11) Hapsari, S.; Yohed, I.; Kristianita, R.A.; Jadid, N.; Aparamarta, H.W.; Gunawan, S. Phenolic and flavonoid compounds extraction from *Calophyllum inophyllum* leaves. *Arab. J. Chem.* 15, 103666 (2022).
12) Prasannakumar, P.; Edla, S.; Thampi, A.D.; Arif, M.; Santhakumari, R. A comparative study on the lubricant properties of chemically modified *Calophyllum inophyllum* oils for bio-lubricant applications. *J. Clean. Prod.* 339, 130733 (2022).
13) Mishra, B.; Routara, B. Evaluation of technical feasibility and environmental impact of *Calophyllum inophyllum* (Polanga) oil based bio-dielectric fluid for green EDM. *Measurement* 159, 107744 (2020).
14) Vishali, K.; Rupesh, K.; Prabakaran, S.; Sudalai, S.; Upputuri, K.B.; Arumugam, A. Development and techno-economic analysis of *Calophyllum inophyllum* biorefinery for the production of biodiesel, biohydrogen, bio-oil, and biochar: Waste to energy approach. *Renew. Energy* 218, 119313 (2023).
15) Cheng, Y.-S.; Wu, J.-H.; Yeh, L.-H. Utilization of *Calophyllum inophyllum* shell and kernel oil cake for reducing sugar production. *Bioresour. Technol.* 212, 338-341 (2016).
16) Arumugam, A.; Shereen, M.F. Bioconversion of *Calophyllum inophyllum* oilcake for intensification of rhamnolipid and polyhydroxyalkanoates co-production by Enterobacter aerogenes. *Bioresour. Technol.* 296, 122321 (2020).
17) Sakthivel, R.; Ramesh, K.; Shameer, P.M.; Purnachandran, R. Experimental investigation on improvement of storage stability of bio-oil derived from intermediate pyrolysis of *Calophyllum inophyllum* seed cake. *J. Energy Inst.* 92, 768-782 (2019).
18) Adenuga, A.A.; Amos, O.D.; Oyekunle, J.A.O.; Umukoro, E.H. Adsorption performance and mechanism of a low-cost biosorbent from spent seedcake of *Calophyllum inophyllum* in simultaneous cleanup of potentially toxic metals from industrial wastewater. *J. Environ. Chem. Eng.* 7, 103317 (2019).
19) Arumugam, A.; Gopinath, K.; Anuse, P.; Shwetha, B.; Ponnusami, V. Kinetic modelling and techno-economic analysis of biodiesel production from *Calophyllum inophyllum* oil. *Biomass Convers. Biorefin.* 9, 363-378 (2019).
20) Kshirsagar, C.M.; Anand, R. Homogeneous catalysed biodiesel synthesis from Alexandrian Laurel (*Calophyllum inophyllum* L.) kernel oil using ortho-phosphoric acid as a pretreatment catalyst. *Int. J. Green Energy* 14, 754-764 (2017).
21) Adenuga, A.A.; Idowu, O.O.; Oyekunle, J.A.O. Synthesis of quality biodiesel from Calophyllum inophyllum kernels through reactive extraction method: Optimization of process parameters and characterization of the products. *Renew. Energy* 145, 2530-2537 (2020).
22) Atabani, A.; Silitonga, A.; Ong, H.C.; Mahlia, T.; Masjuki, H. *et al.* Non-edible vegetable oils: A critical evaluation of oil extraction, fatty acid compositions, biodiesel production, characteristics, engine performance and emissions production. *Renew. Sustain. Energy Rev.* 18, 211-245 (2013).
23) Ajala, S.O.; Betiku, E. Yellow oleander seed oil extraction modeling and process parameters optimization: Performance evaluation of artificial neural network and response surface methodology. *J. Food Process. Preserv.* 39, 1466-1474 (2015).
24) Ibrahim, A.P.; Omilakin, R.O.; Betiku, E. Optimization

of microwave-assisted solvent extraction of non-edible sandbox (*Hura crepitans*) seed oil: A potential biodiesel feedstock. *Renew. Energy* **141**, 349-358 (2019).
25) Oladipo, B.; Betiku, E. Process optimization of solvent extraction of seed oil from *Moringa oleifera*: An appraisal of quantitative and qualitative process variables on oil quality using D-optimal design. *Biocatal. Agric. Biotechnol.* **20**, 101187 (2019).
26) Jisieike, C.F.; Betiku, E. Rubber seed oil extraction: Effects of solvent polarity, extraction time and solid-solvent ratio on its yield and quality. *Biocatal. Agric. Biotechnol.* **24**, 101522 (2020).
27) Ajala, E.O.; Aberuagba, F.; Olaniyan, A.M.; Onifade, K.R. Optimization of solvent extraction of shea butter (*Vitellaria paradoxa*) using response surface methodology and its characterization. *J. Food Sci. Technol.* **53**, 730-738 (2016).
28) Myers, R.H.; Montgomery, D.C.; Anderson-Cook, C. *Response surface methodology: Product and process optimization using designed experiments*. John Wiley & Sons, New York (2009).
29) AOAC. *Official methods of analyses of the Association of Official Analytical Chemists*, 15th Ed., Association of Official Analytical Chemists, Washington DC, USA (1990).
30) Betiku, E.; Odude, V.O.; Ishola, N.B.; Bamimore, A.; Osunleke, A.S.; Okeleye, A.A. Predictive capability evaluation of RSM, ANFIS and ANN: A case of reduction of high free fatty acid of palm kernel oil via esterification process. *Energy Convers. Manag.* **124**, 219-230 (2016).
31) Joglekar, A.; May, A. Production excellence through design of experiment. *Cereal Foods World* **32**, 857-868 (1987).
32) Sharma, A.; Kodgire, P.; Kachhwaha, S.S. Biodiesel production from waste cotton-seed cooking oil using microwave-assisted transesterification: Optimization and kinetic modeling. *Renew. Sustain. Energy Rev.* **116**, 109394 (2019).
33) Noshadi, I.; Amin, N.; Parnas, R.S. Continuous production of biodiesel from waste cooking oil in a reactive distillation column catalyzed by solid heteropolyacid: optimization using response surface methodology (RSM). *Fuel* **94**, 156-164 (2012).
34) Bhuiya, M.; Rasul, M.; Khan, M.; Ashwath, N.; Mofijur, M. Comparison of oil extraction between screw press and solvent (*n*-hexane) extraction technique from beauty leaf (*Calophyllum inophyllum* L.) feedstock. *Ind. Crops Prod.* **144**, 112024 (2020).
35) Zhang, Q.-W.; Lin, L.-G.; Ye, W.-C. Techniques for extraction and isolation of natural products: A comprehensive review. *Chinese Medicine* **13**, 1-26 (2018).
36) Betiku, E.; Adepoju, T.F.; Omole, A.K.A.; Aluko, S.E. Statistical approach to the optimization of oil from beniseed (*Sesamum indicum*) oil seeds. *Journal of Food Science and Engineering* **2**, 351-357 (2012).
37) Mohd Shamsuddin, N.; Yusup, S.; Ibrahim, W.; Bokhari, A.; Chuah, L. Oil extraction from *Calophyllum inophyllum* L. via soxhlet extraction optimization using response surface methodology (RSM). A paper presented at the 10th Asian Control Conference (10th ASCC). 31 May-3 June, 2015 at Sutera Harbour Resort, Kota Kinabalu, Sabah, Malaysia. *2015 10th Asian Control Conference (ASCC)* (2015). doi: 10.1109/ASCC.2015.7244791
38) Atabani, A.; Mahlia, T.; Badruddin, I.A.; Masjuki, H.; Chong, W.; Lee, K.T. Investigation of physical and chemical properties of potential edible and non-edible feedstocks for biodiesel production, a comparative analysis. *Renew. Sustain. Energy Rev.* **21**, 749-755 (2013).
39) Onyeike, E.N.; Acheru, G.N. Chemical composition of selected Nigerian oil seeds and physicochemical properties of the oil extracts. *Food Chem.* **77**, 431-437 (2002).
40) Ikyenge, B.A.; Ageh, J.T.; Anhwange, B.A.; Nyiatgher, D.T. Synthesis and Characterization of vegetable oil-based polyol from *Thevieta peruviana* seed oil *International Journal of Modern Organic Chemistry* **1**, 55-59 (2012).
41) Belagur, V.K.; Chitimi, V.R. Few physical, chemical and fuel related properties of *Calophyllum inophyllum* linn (honne) oil and its blends with diesel fuel for their use in diesel engine. *Fuel* **109**, 356-361 (2013).
42) Ramadhas, A.S.; Jayaraj, S.; Muraleedharan, C. Biodiesel production from high FFA rubber seed oil. *Fuel* **84**, 335-340 (2005).
43) Ghadge, S.V.; Raheman, H. Biodiesel production from mahua (*Madhuca indica*) oil having high free fatty acids. *Biomass Bioenergy* **28**, 601-605 (2005).
44) Sarin, R.; Sharma, M.; Sinharay, S.; Malhotra, R.K. Jatropha–palm biodiesel blends: an optimum mix for Asia. *Fuel* **86**, 1365-1371 (2007).
45) César, Aldara da Silva. A competitividade da produção de biodiesel no Brasil: uma análise comparativa de mamona, dendê e soja. 2012. 260 f. Tese (Doutorado em Ciências Exatas e da Terra) - Universidade Federal de São Carlos, São Carlos (2012).
46) Sahoo, P.; Das, L. Process optimization for biodiesel production from Jatropha, Karanja and Polanga oils. *Fuel* **88**, 1588-1594 (2009).
47) Rajendran, N.; Gurunathan, B. Optimization and technoeconomic analysis of biooil extraction from *Calophyllum inophyllum* L. seeds by ultrasonic assisted solvent oil extraction. *Ind. Crops Prod.* **162**, 113273 (2021).
48) Safar, M.; Bertrand, D.; Robert, P.; Devaux, M.F.; Genot, C. Characterization of edible oils, butters and

margarines by Fourier transform infrared spectroscopy with attenuated total reflectance. *J. Am. Oil Chem. Soc.* 71, 371-377 (1994).

49) Coates, J. Interpretation of Infrared Spectra: A Practical Approach. in *Encyclopedia of Analytical Chemistry* (Meyers, R.A. ed.), John Wiley and Sons Ltd., Chichester (2000).

50) Mofijur, M.; Atabani, A.; Masjuki, H.H.; Kalam, M.A.; Masum, B.M. A study on the effects of promising edible and non-edible biodiesel feedstocks on engine performance and emissions production: A comparative evaluation. *Renew. Sustain. Energy Rev.* 23, 391-404 (2013).

51) Sahoo, P.; Das, L.; Babu, M.; Naik, S. Biodiesel development from high acid value polanga seed oil and performance evaluation in a CI engine. *Fuel* 86, 448-454 (2007).

52) Ramaraju, A.; Kumar, T.A. Biodiesel development from high free fatty acid punnakka oil. *ARPN J. Eng. Appl. Sci* 6, 1 (2011).

53) Silitonga, A.; Masjuki, H.; Ong, H.C.; Kusumo, F.; Mahlia, T.; Bahar, A. Pilot-scale production and the physicochemical properties of palm and *Calophyllum inophyllum* biodiesels and their blends. *J. Clean. Prod.* 126, 654-666 (2016).

54) Fattah, I.R.; Kalam, M.; Masjuki, H.; Wakil, M. Biodiesel production, characterization, engine performance, and emission characteristics of Malaysian Alexandrian laurel oil. *Rsc Adv.* 4, 17787-17796 (2014).