Optimization of Solvent Extraction of Oil from Sandbox Kernels (*Hura Crepitans* L.) by a Response Surface Method

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**ABSTRACT**

The use of vegetable oil as feedstock for biodiesel production is controversial as a result of the challenges of a food-fuel crisis associated with the use of edible oils for biodiesel production and use of arable land for energy feedstock generation. This work, therefore, focused on the extraction of oil from non-edible seed such as sandbox seed using a solvent extraction method, evaluation of optimal conditions for oil extraction from sandbox, and testing the fuel properties of extracted sandbox oil. A Box-Behnken design of response surface methodology (RSM) with 17 experimental runs was used to investigate the optimum conditions for the extraction, and the selected variables were effective seed/solvent ratio (0.04, 0.06, 0.08 g/ml), extraction temperature (65, 60, 75°C), and extraction time (3, 5, 7 h). Selected fuel properties (specific gravity, viscosity, cloud point, pour point, density, and refractive index) of the extracted oil were determined according to American Society for Testing Materials (ASTM) standards.

The optimum oil yield (63.4%) was obtained at the seed/solvent ratio, extraction temperature, and extraction time of 0.05 g/ml, 68.13°C, and 5 h, respectively. The viscosity at 30°C, specific gravity, density, cloud point, pour point, and refractive index of the extracted oil were 4.55 mm²/s, 0.91, 910 kg/m³, 5.9°C, −1.0°C, and 1.4683, respectively. Thus the result from this research work has established the optimal conditions for solvent extraction of oil from sandbox seed. The fuel properties of the sandbox oil show that it is potentially suitable to produce biodiesel that can be used to power internal combustion engines.

**1. Introduction**

An increase in the world population of oil seeds in the last three decades has an increasing demand for oilseed products and their byproducts.¹ This has led to the need to search for more oilseeds, which can be used as raw materials for industrial purposes and which can establish a new pathway from prospective oil-producing seeds. Ogunkunli² reported that in Africa, where most countries are struggling with development, there is a need to replace industrial oil-based seeds mostly used as food with non-edible ones. The replacement will be helpful in reducing the challenges of food competition as well as industrial uses of the same oil such as groundnut oil, moringa oil, soya oil, palm kernel oil, corn oil, and other edible oils. Sandbox (*Hura crepitans*), also known as possum wood, and jabillo is an evergreen tree of the spurge family (Euphorbiaceae). It is usually found in tropical regions of North and South America in the Amazon rainforest. The seeds are enclosed in a hard, protective coat, which usually and suddenly splashes open and scatters when the seeds are well dried. It is commonly planted in the cities and villages of the southwestern, north central (Markurdi and Kogi), and southern (Edo State) parts of Nigeria, mostly for shade. It is known as *Odan Mecca* by the Kabba people of Kogi State, Nigeria, and as *Aroyin* by the Ijesha people of Osun State, Nigeria.³ Apart from being seen as a prospect for biodiesel production, sandbox may possibly have edible uses, industrial uses, and pharmaceutical uses. A lot of researchers⁴,⁵,⁶ have worked on the extraction of oil from sandbox kernels, but the effect of some process parameters/conditions for this extraction requires more extensive study to determine the optimal extraction conditions. Response surface methodology has been described as an effective approach in studying the effects of individual variables and their interactions on response variables. It has been used extensively on extraction of oil from edible and non-edible oils from different sources. Therefore, this study investigates the effects of some process parameters on the extraction of oil from sandbox kernels using response surface methodology.

The quality of the extracted raw sandbox oil was evaluated by carrying out some selected physicochemical properties. The fatty acid composition (wt%) of *Hura crepitans* seed oils was reported by Adewuyi et al.⁷ The composition is shown in Table 1. The dominant fatty acid in the oil as reported contains C18:2 (52.8 ± 0.10%).

Oil can be obtained from oilseeds by using mechanical expression or solvent extraction methods or a combination of both. In mechanical expression, hydraulic and screw presses are usually employed. Solvent extraction is capable of removing virtually all of the oil available in the oil seed.⁴

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2. Materials And Methods

2.1 Materials

**Sandbox (Hura crepitans L.) seeds**

Sandbox fruits, a natural precursor, were procured at the premises of the Department Agricultural and Environmental Engineering University of Ibadan Oyo State, Nigeria. Both matured and dried fruit of this tree were harvested in bulk quantity. The pods of the seeds were removed naturally by subjecting the seeds to sun drying, after which the pods break on their own; this is preferable to the use of a mortar and pestle. The obtained dried seeds were parboiled to reduce the hardness of the seed shell. The sandbox kernel was obtained by removing the shell of the parboiled seed by hand. The kernel was sundried at atmospheric temperature for about two weeks and then ground into fine particles.

2.2 Experimental design

The experiment was designed using Design Expert version 6.0.8, where a Box-Behnken experimental design was employed in order to optimize the extraction of oil from sandbox kernels. The experiment was designed on three levels, three factors that generated 17 experimental runs. The three independent factors are extraction time, extraction temperature, and seed/solvent ratio. The summary of this is as shown in Table 2.

2.3 Extraction of oil from sandbox (Hura crepitans L.) kernels using solvent extraction

The extraction of oil was carried out at the laboratory of the Department of Chemical Engineering, Ladoke Akintola University of Technology, Oyo state, Nigeria, using the method described by Afolabi et al.8 The extraction was done with a soxhlet apparatus of 250 cm³ capacity using n-hexane as the solvent. The extraction was done by using a prepared sample of 10–20 g sandbox kernel, extraction time of 3 to 7 h, and extraction temperature between the ranges of 65°C and 75°C, as given in Table 2.

The experimental runs were carried out according to the experimental runs generated from Design Expert. The solvent used was recovered at every experimental run through a distillation process, and the actual oil obtained was weighed. The experiment was repeated for other parameters, and the percentage yield was calculated.

The oil yield for the extraction were determined by using the equation:

\[
O.Y = \frac{M_0}{M_S} \times 100 \tag{1}
\]

where:

- \(O.Y\) = oil yield (%)
- \(M_0\) = mass of oil extracted (g)
- \(M_S\) = mass of the Hura crepitans seed (g)

A schematic diagram showing the components of the experimental apparatus for oil extraction is shown in Figure 1.

2.4 Determination of some selected physicochemical properties

Evaluation of some selected physicochemical properties of the sandbox oil was carried out using ASTMD 6751 standard procedures. The selected properties are refractive index, viscosity, specific gravity, pour point, and cloud point.

2.5 Optimization studies using response surface methodology

Response surface methodology has been used to study the optimization of chemical processes and products.9,10 Thus, response surface methodology was used in this study to investigate the optimum process parameters for the extraction of oil from sandbox seed using solvent extraction. The factors considered are solvent ratio, extraction temperature, and extraction time, among others, for optimal oil yield. A three-level, three-factor Box-Behnken design (BBD) was employed using Design Expert 6.0.8 software to examine the optimum conditions of extraction of oil from sandbox seed using solvent extraction. The generated runs for the BBD investigated in this work consist of 17 experimental runs.

3. Results and Discussion

3.1 Result of oil yield from the extraction of oil from sandbox (Hura crepitans L.) kernels

The result of the solvent extraction of oil from sandbox oil is presented in Table 3. The yield for each run of the experiment was determined using Equation 1. The varying oil yield values are indications that the extraction parameters considerably affect the oil yield. It was observed that the yields obtained compared well with the predicted yield by the Design Expert software. The maximum oil yield of 62% was obtained from the extraction of the corresponding seed/solvent ratio of 0.06, extraction

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Table 1. Fatty acid composition (wt%) of Hura crepitans seed oils.

| Fatty acids | Hura crepitans |
|------------|---------------|
| 16:0       | 12.20 ± 0.20  |
| 16:1       | 0.10 ± 0.00   |
| 18:0       | 5.1 ± 0.30    |
| 18:1       | 27.2 ± 0.20   |
| 18:2       | 52.8 ± 0.10   |
| 18:3       | 1.8 ± 0.10    |
| 20:0       | 0.2 ± 0.10    |
| 20:1       | 0.3 ± 0.10    |
| 22:0       | 0.3 ± 0.10    |
| Unsaturated| 82.2 ± 0.20   |
| Saturated  | 17.80 ± 0.20  |

Values are mean ± standard deviation of triplicate determinations.

Table 2. Summary of the experimental design.

| Factor | Name           | Unit | Type | Level | -1  | 0   | 1   |
|--------|----------------|------|------|-------|-----|-----|-----|
| A      | Seed/solvent ratio | g/ml | Numeric | 0.04  | 0.06 | 0.08 |
| B      | Extraction temp | °C   | Numeric | 65.00 | 70.00 | 75.00 |
| C      | Extraction time | (h)  | Numeric | 3.00  | 5.00 | 7.00 |
temperature of 70°C, and extraction time of 5 h. The maximum value obtained for the yield seemed to be slightly high than 58.87% reported by Muhammed et al.\textsuperscript{11} using solvent extraction and n-hexane as a solvent. Also, the percentage oil yield compared well with the yield obtained from sandbox seed extraction being reported to have an oil yield of 53.61%.\textsuperscript{4} It may be suggested that the variation in the oil yield of sandbox may be due to the differences in the variety of the fruit, the species, and the environmental conditions. This is an indication that the oil yield from the extraction of oil from sandbox kernels was affected by the process parameters or conditions.

3.2 Statistical analysis of data for extraction of oil from sandbox (Hura crepitans) seeds

The statistical analysis for oil extraction from sandbox seeds was done using analysis of variance (ANOVA). Table 4 shows the ANOVA results of oil extraction. The multiple regression analysis of the experimental data gives a second-order polynomial equation, which was modified to discard the insignificant B, C, $B^2$, and BC model terms. The reduced quadratic model developed depicts the interaction between the dependent oil yield (Y) and the coded values of the independent variables A, B, and C (seed/solvent ratio, extraction temperature, and extraction time). This is shown in equation 2.

$$Y = 60.29 - 3.01A - 0.68B - 1.57C - 3.20A^2 - 1.89B - 6.86C^2 + 5.51AB + 3.96AC - 0.28BC$$  

where Y represents response variable oil yield measured in %.

The significance and adequacy of the model was tested by using ANOVA. It was observed from the table that the linear interaction effects are due to the Coded Factor A, corresponding to seed/solvent ratio, which is a significant factor. The quadratic effects of solvent ($A^2$), quadratic effects of extraction time ($C^2$), seed/solvent ratio and extraction temperature

\begin{table}[h]
\centering
\caption{Experimental design layout using Box-Behnken design.}
\begin{tabular}{cccccc}
\hline
Std & Runs & Factor A Seed/solvent Ratio (g/ml) & Factor B Extraction Temperature (°C) & Factor C Extraction time (h) & Response Oil yield (%) & Predicted Oil Yield (%) \\
\hline
9 & 1 & 0.06 & 65.0 & 3.0 & 53.5 & 53.50 \\
7 & 2 & 0.04 & 70.0 & 7.0 & 48.5 & 47.31 \\
2 & 3 & 0.08 & 65.0 & 5.0 & 48.15 & 46.96 \\
11 & 4 & 0.06 & 65.0 & 7.0 & 50.12 & 51.31 \\
8 & 5 & 0.08 & 70.0 & 7.0 & 49.6 & 49.60 \\
10 & 6 & 0.06 & 75.0 & 3.0 & 55.8 & 54.61 \\
1 & 7 & 0.04 & 65.0 & 5.0 & 52.5 & 52.12 \\
4 & 8 & 0.08 & 75.0 & 5.0 & 59.9 & 59.90 \\
16 & 9 & 0.06 & 70.0 & 5.0 & 60.6 & 60.28 \\
5 & 10 & 0.04 & 70.0 & 3.0 & 51.4 & 51.80 \\
3 & 11 & 0.04 & 75.0 & 5.0 & 51.2 & 52.39 \\
14 & 12 & 0.06 & 70.0 & 5.0 & 57.7 & 60.23 \\
13 & 13 & 0.06 & 70.0 & 5.0 & 61.0 & 60.28 \\
12 & 14 & 0.06 & 75.0 & 7.0 & 49.0 & 49.00 \\
17 & 15 & 0.06 & 70.0 & 5.0 & 60.1 & 60.28 \\
6 & 16 & 0.08 & 70.0 & 3.0 & 44.0 & 45.19 \\
15 & 17 & 0.06 & 70.0 & 5.0 & 62.0 & 60.28 \\
\hline
\end{tabular}
\end{table}

\begin{figure}[h]
\centering
\includegraphics[width=\textwidth]{fig1.png}
\caption{Schematic components of the experimental apparatus for oil extraction.}
\end{figure}
(AB), and seed/solvent ratio and extraction time are all significant being less than 0.05, while the values greater than 0.05 means not significant. The ANOVA result shows that the factors B, C, B², and BC are not significant.

The interaction effects of seed/solvent ratio, time, and temperature on the yield were studied using the interaction plot and 3D surface plot of RSM. From Figure 2 the combined effect of time and seed/solvent ratio was studied using the interaction plot of RSM, and it was observed that the oil yield increased with increase in time and seed/solvent ratio. Also, it can be deduced from Figure 3 that increase in seed/solvent ratio and extraction temperature favors the extraction rate as much solvent is needed to ensure that vapor rose through the vertical monitor tube into the condenser at the top just as the solvent boiled. However, the oil yield was observed to decrease with higher temperature, particularly at the temperature higher than the boiling temperature of the solvent. This is because at this temperature higher than the boiling temperature of the solvent, evaporation of the solvent will take place as a result of the increased temperature. The behavior of time, temperature, and seed/solvent ratio in this study is similar to previous work of Afolabi et al., who worked on optimization of solvent extraction of oil from Parinari polyandra. It was reported that the effect of the decrease in oil yield at a higher temperature is more prominent in hexane than when another solvent is used. This may be due to its low boiling point of 67ºC when compared with petroleum ether. This relatively lower temperature advantage of n-hexane may be indicative of a more economical solvent when considering heating cost among other requirements for solvent extraction.

From Table 4, standard deviation of 1.68, mean of 53.73, C.V. of 3.13, R² of 0.9684, adj. R² of 0.9114, and adeq. precision of 11.264 were obtained for biodiesel yield. The fitness of the polynomial model was expressed by the coefficient of determination of R² and the coefficient of adjusted R², which were obtained as 0.9684 and 0.9114, respectively. It is suggested that these values should be at least 0.80 for the good fit of the model. Therefore, the adjusted R² value of 0.9684 and the adjusted R² value of 0.9114 indicate that the regression model is acceptable. The lack of fit value of 1.51

| Source Model | Sum of Squares | Degree of Freedom | Mean Square | F Value | Prob> F  |
|--------------|---------------|------------------|-------------|---------|---------|
| Model        | 433.45        | 9                | 48.16       | 17.01   | 0.0031  |
| A            | 24.20         | 1                | 24.20       | 8.55    | 0.0329  |
| B            | 2.24          | 1                | 2.24        | 0.79    | 0.4149  |
| C            | 11.79         | 1                | 11.79       | 4.17    | 0.0967  |
| A²           | 21.07         | 1                | 21.07       | 7.44    | 0.0414  |
| B²           | 11.13         | 1                | 11.13       | 3.93    | 0.1042  |
| C²           | 146.68        | 1                | 146.68      | 51.81   | 0.0008  |
| AB           | 52.05         | 1                | 52.05       | 18.38   | 0.0078  |
| AC           | 26.95         | 1                | 26.95       | 9.52    | 0.0273  |
| BC           | 0.31          | 1                | 0.31        | 0.11    | 0.7528  |
| Residual     | 14.16         | 5                | 2.83        |         |         |
| Lack of Fit  | 3.89          | 1                | 3.89        | 1.51    | 0.2859  |
| Pure Error   | 10.27         | 4                | 2.57        |         |         |
| Cor Total    | 447.60        | 14               |             |         |         |
| Std. Dev.    | 1.68          |                  |             |         |         |
| Mean         | 53.73         |                  |             |         |         |
| C.V.         | 3.13          |                  |             |         |         |
| PRESS        | N/A           |                  |             |         |         |

Table 4. Analysis of variance (ANOVA) for extraction of oil from sandbox (partial sum of squares).
implies nonsignificance; this is relative to the pure error. This was desirably low for the model, which shows adequate representation of the interaction by the model. Nonsignificant lack of fit of the model is good as the model could be used for theoretical prediction of the oil extraction. It can be deduced from the F-value of 17.01 with a corresponding low probability value of 0.0031, which is less than 0.05, that the model terms are significant. In this case, the adequate precision value (P adeq = 11.264) is greater than 4, which is desirable for the model showing an adequate signal-to-noise ratio of the model.

Table 5 shows the assessment of experimental errors and the confidence interval (CI) of the experimental variables. The standard errors are analyzed based on the differences between the predicted oil yields and the experimental values recorded as shown in Table 4.

3.2.1 Effect of the seed/solvent ratio and extraction time on the oil yield from sandbox

The 3D surface plot interaction from Design Expert presented in Figure 2 represents the effects of seed/solvent ratio and extraction time. Their reciprocal interactions on the oil yield for extraction were studied. It was observed that increase in seed/solvent ratio favors the extraction rate as much solvent is needed for extraction, while the oil yield increases gradually as extraction time increases until the boiling temperature of the solvent is reached, when the yield starts declining. The curvature nature of the surface plot in Figure 2 depicts a mutual interaction between extraction time and seed/solvent ratio.

3.2.2 Effect of seed/solvent ratio and extraction temperature on the oil yield from sandbox seed

The effect of seed/solvent ratio and extraction temperature was studied using the 3D surface plot in Figure 3, which depicts the interaction between seed/solvent ratio and extraction temperature. The higher the temperature, the higher the extraction rate, until the temperature rises above the boiling temperature of the solvent, when further increment of the temperature led to a decrease in the oil yield. It can be deduced from the analysis of variance (ANOVA) results in Table 4 that the combined effects of seed/solvent ratio and extraction temperature have a significant effect on the oil yield from sandbox seed.

3.2.3 Effect of extraction time and extraction temperature on the oil yield from sandbox seed

From Figure 4, the elliptical nature of the contour plots indicated that interaction between the extraction temperature and time had a direct significant effect on the yield of oil. It was noticed that increase in time favored the extraction of oil until it got close to the moderate reaction time. Increased reaction time above this level tends to produce lower oil yields. The oil yield was observed to decrease with higher temperature particular at the temperature higher than the boiling point of the solvent. This is because at this temperature higher than the boiling temperature of the solvent, evaporation of the solvent will take place as a result of the increased temperature. The combined effect of these two reaction variables at high-level experimental process will obviously result in a decrease in oil yield as recorded in Table 3.
3.3 Optimization of processing parameters for maximum oil yield

Determination of the optimum yield was based on numerical optimization using Design Expert software. The maximum recovery of oil was obtained at the solvent ratio, extraction temperature, and extraction time of 0.05 g/ml, 68.43ºC, and 4.62 h, respectively, with the oil yield comparing well with literature. The surface plot of the optimum value is illustrated in Figure 5. Under these optimum conditions, the oil recovery was predicted as 63.4% with desirability of 1.000%. These optimum conditions compared well with the experimental value, which gives the optimum oil yield of 62.0%, a value slightly lower than the predicted value of 63.4%. To verify and confirm the agreement between the results obtained from the model, the optimum conditions obtained were taken to the laboratory to validate the prediction of the model. A confirmatory experiment was done three times using the optimal conditions, and the average sandbox oil yield obtained was 61.9%. The result obtained from experiment has established the optimal conditions for solvent extraction of oil from sandbox seed using response surface methodology for optimization.

3.4 Physicochemical properties of the extracted sandbox oil

The extracted sandbox oil was analyzed for some selected physicochemical properties so as to determine the quality of the oil. The result for the analysis is shown in Table 6. The oil has a viscosity of 4.55 mm²/s, specific gravity of 0.91,
cloud point of 5.9°C, pour point of -1.0°C, golden yellow color, refractive index of 1.4683 at 30°C, and acid value of 2.771 mgKOH/g. The results are in close agreement with findings of Okolie et al.\textsuperscript{4} and Oniya et al.,\textsuperscript{13} who worked on sandbox, except for a little variation that may be due to different process conditions. The result of the specific gravity and refractive index was in agreement with 0.88 and 1.464 (25°C), reported by Afolabi et al.,\textsuperscript{8} oil extraction from \textit{Parinari polyandra}. The result of the specific gravity also conforms well to 0.88, reported by Betiku and Adepoju,\textsuperscript{14} in extraction of oil from sorrel seed. The results obtained from each of these properties evaluated were found to be in agreement with the ASTM D6751 standard for biodiesel. This then suggests the viability of sandbox (\textit{Hura crepitans}) oil as a prospect for biodiesel production.

### 4. Conclusion

Based on the findings of this research work (the result of the extraction of oil, physiochemical properties, and optimization process), sandbox (\textit{Hura crepitans}) seed oil has potential as a source of fuel for diesel engines. The optimal conditions for extraction of oil from sandbox were given as a seed/solvent ratio of 0.05 g/ml, extraction temperature of 68.43°C, and extraction time of 4.62 h, with the predicted oil yield as 63.4%.

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### Table 6. Some selected physicochemical properties of the extracted sandbox oil.

| Physicochemical properties          | Result   | ASTM Standard |
|-------------------------------------|----------|---------------|
| Specific gravity                    | 0.91     | 0.87-0.98     |
| Viscosity at room temp (mm²/s)      | 4.45     | 1.90-6.0      |
| Cloud point (°C)                    | 5.9     | Varies        |
| Pour point (°C)                     | -1.0     | -15-13        |
| Color                               | Golden yellow |          |
| Refractive index at room (30°C)     | 1.4683   |               |
| Oil yield (%)                       | 62       |               |
| Acid value (mgKOH/g)                | 2.771    | < 10          |

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Plate 1. Sandbox flower, ripe fruit, seeds, and kernel.

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