

Optimization of Solvent Oil Extraction from Sandbox (*Huracrepitans Linn.*) Seeds

ABSTRACT

Due to increasing demand for food, industrial applications and biodiesel production on vegetable oils, optimization of oil extraction processes has become imperative to obtain maximum oil yield from vegetable oil feedstocks. The effect of process factors: moisture content, roasting temperature, and time, extraction temperature and time on oil yield from sandbox seed by solvent extraction was investigated using a 5 × 5 Central Composite Rotatable (Experimental) Design of Response Surface Methodology. Results obtained were analyzed using Analysis of Variance (ANOVA) and SPSS statistical tool at ($p = 0.05$). Optimum conditions predicted were validated by experiments. The American Oil Chemists' Society AOCs 5-04 standard procedure for solvent extraction was used in the experiment. The oil yield from the sandbox seed ranged from 20.9-53.6%, and was increased at the range (6-10%) moisture content, (80-100°C) roasting temperature, (5-15 min) roasting time, (90-100°C) extraction temperature and (90-150 min) extraction time. The optimum oil yield of 53.6% was obtained at the processing conditions of 10.0% moisture content, 95°C roasting temperature, 15 min roasting time, 65°C extraction temperature and 90 min extraction time. Mathematical models to predict sandbox seed oil yield at varying process conditions were developed with an R^2 (0.81). The optimum extractable oil yield of 57.10% was predicted for sandbox seed at processing conditions of 10.25% moisture content, 98.72°C roasting temperature, 17.63 min roasting time, 67.01°C extraction temperature and 96.6 min extraction time. The study results provide data for equipment and process designs for oil extraction from sandbox and other oilseeds.

Keywords: *Huracrepitans*, Oil extraction, Oilyield, Process optimization, Solvent extraction

1. INTRODUCTION

Sandbox (*Huracrepitans* Linn.) tree is a dicotyledon belonging to the Euphorbiaceae (spurge) family. It is an evergreen perennial tree of origin of the tropical zones of the Amazon

rainforest of North and South America [4]. The sandbox seed is a source of oil [13], and may have edible, industrial application and pharmaceutical potentials. It is however grouped under underutilized species of plant [4]. According to [24], in many areas of the globe, the tree serves as shade trees as a result of its large spreading branches. Sandbox seeds have been reported as an underutilized plant in Nigeria, which are planted as shade trees in villages and towns, as the seeds are discarded as waste since they have no specific use at present [3]. Oils and fats are of great importance globally due to their extensive application in homes and industries. They form a very important part of the human diet as over ninety percent of the world production of vegetable oils, animal fats and marine oils are utilized directly as food or as ingredients in food products [19]. Global industrialization and pollution from fossil fuel combustion has led to increasing demand for environmentally acceptable energy sources [28, 10]. The situation triggered the quest into the exploration and utilization of more vegetable oil sources as feedstocks for oil and fat to meet the ever increasing global demands. The production of oil from oilseeds is a lucrative endeavor, thus, investigations into ways to improve the extraction process for better oil yield and quality from seeds and other materials is on the increase. Oil extraction from oil seeds and other biological materials are carried out by methods such as: solvent extraction, mechanical pressing, aqueous, enzymatic and supercritical fluid extraction [17]. Solvent extraction processes increase oil recovery from lipid cells and have made it economically attractive to process seeds, nuts and other materials with relatively low oil content. Solvent extraction can achieve oil recovery of up to 98% [27]. Solvent extraction involves dissolving soluble constituents from solid materials by means of suitable solvents. Crude liquids such as acetone, ether, chloroform, petroleum, spirit, carbon tetrachloride, diethyl ether and carbon tetra chloride are all used in oil extraction from oil bearing materials [25]. Research into optimum process conditions for maximum oil yield by solvent extraction method has been carried out on so many oilseeds: sesame seed [23, 18]; rubber seed [11]; soybean seed [26]; dried Bitter Gourd seed [41]; unsieved rice bran and sieved rice bran respectively [39]; sunflower kernels [40] and grated coconut [22]; Neem (*Azadirachta indica*) seed [12]; Sorrel (*Hibiscus sabdariffa*) seed [16]; almond seed [7]; Dika nut [1]; groundnut [33]; Shea kernel [34]; African star apple seed [5]; *Moringa* seed [2].

Oil extractions by solvent extraction method from sandbox seed have been reported by [32, 29, 4, 38, 31, 37].

The quality and quantity of extractable oil from oilseeds is dependent on: the state of the seeds before extraction, the extraction method employed, effectiveness of the extraction machine or procedure. Other parameters that can be juggled with to influence the oil yield include seed and machine parameters such as; the feedstock moisture content, particles

size, heating temperature and time, and applied pressure and also duration of extraction [30]. Data for the best extraction conditions for solvent oil extraction from sandbox seed is however scarce. To quantify and predict oil yield from sandbox seed by solvent extraction relative to process factors, the Response Surface Methodology (RSM) was employed. According to [21], a process where many input factors affect an outcome has been usually done by varying one factor while other factors are kept constant. This procedure basically does not capture the inter-relationship existing amongst the factors and hence it may not accurately predict the best combination of interaction of factors that gives the best outcome of the process. Thus, the Response Surface Methodology (RSM) was developed as a useful statistical tool for process optimization utilizing experimental designs such as Central Composite Design (CCD), Box-Behnken design and D-optimal design. Better than the conventional method, the RSM predicts combination of conditions with minimal number of experiments and also develops mathematical extraction (s) showing the relationships between the response(s) and the factors. The main advantage of RSM is the reduction in the number of experimental runs required to give sufficient information for statistically acceptable outcome, and it is a faster and less expensive approach of obtaining research results than the common methods [43, 42]. The present work investigated how process factors: moisture content, treating temperature and time, extraction temperature and time influence solvent oil extraction and oil yield from sandbox seed using the RSM.

2. MATERIAL AND METHODS

Design of experiment

Variations of oil extraction processing parameters have been observed to influence oil yield. The process of oil extraction from sandbox seed by solvent extraction was carried out by varying the moisture content, roasting temperature and time, extraction temperature and time. A 5x5 factorial Central Composite Rotatable Design (CCRD) of Response Surface Methodology developed by [15] was used for the experiment. The range of moisture content used in the experiment was varied from the initial moisture content of the mature sandbox seed. Due to lack of data on effects of process variables on sandbox oil yield by solvent extraction, information from studies on oil extraction from other oilseeds was used to carry out preliminary investigations on the sandbox seed. The preliminary results informed the range of process factors selected for the experiment. The experimental values were; moisture content, mc (4, 6, 8, 10 and 12% wet-basis); roasting temperature, r_{tp} (80, 85, 90, 95 and 100°C) and time, r_{tm} (0, 5, 10, 15 and 20 min); extraction temperature, ϵ_{tp} (60, 65, 70, 75 and 80°C) and time, ϵ_{tm} (60, 90, 120, 150 and 180 min).

Preparation of Samples

About 100 kg of mature sandbox fruits were collected under the trees in Uyo metropolis, Akwalbom State. The fruits were cracked to remove the seeds and the seeds peeled to get the kernels as shown in Fig. 1.



Fig. 1. Sandbox seedprocessing

Moisture content determination

Initial moisture content of the sandbox seeds was determined using American Society of Agricultural and Biological Engineers (ASABE) standard for oven drying method as adopted by [20, 36] for Moringa and African star apple seeds respectively. Equation 1 below was used to calculate the moisture content (wet-basis).

$$MC(\%w. b) = \frac{W_i - W_f}{W_i} \times 100 \quad (1)$$

W_i = initial sample weight and W_f = final sample weight

1 kg each of the samples were subjected to 4, 6, 8, 10 and 12% wet basis moisture content respectively using Equation 2 as adopted by [19].

$$Q = \left(\frac{100 - S_i}{100 - S_d} - 1 \right) \times W_s \quad (2)$$

Q = quantity of required moisture to be absorbed (ml); S_i = initial sample moisture (%wb); S_d = required sample moisture (%wb); W_s = weight of sample (g)

The conditioned samples were wrapped up to avoid moisture loss and stored in a desiccator to retain them at the conditioned moisture content for the experiment.

Experimental Procedure

From the already conditioned samples of the sandbox seed at 4, 6, 8, 10, 12% wb moisture content, the various experiments were conducted using 500 g weight samples. A hotplate was used for roasting the sandbox seed samples. The various roasting temperature levels of 80, 85, 90, 95 and 100°C were achieved by regulating the hotplate temperature. A frying pot was placed on top of the hotplate, and a digital thermometer probe was used to check the pot temperature until the required temperatures were obtained before pouring the sandbox seed samples for frying. A stopwatch was used to time the roasting periods for 0, 5, 10, 15 and 20 min respectively. Afterwards, the oil extraction was carried out according to AOCS 5-

04 standard procedure. A six-in-one Soxhlet apparatus (Fig. 2) was used for the oil extraction. The Soxhlet method is a semi-continuous process used for extraction of lipids from foods. The procedure utilizes repeated extraction (reflux) as heated organic solvent flow across oil and fat. 500 g finely ground sandbox seed samples were tied in filter papers and placed in a porous cellulose thimble. The thimble was then placed in an extraction chamber which is being suspended above a flask containing the solvent (standard grade petroleum ether) and below a condenser. Heat was applied to the flask and the solvent evaporates and moves through the ground sandbox seed samples in the extraction chamber where it picks up the lipid as it moves to the condenser where it was converted into liquid and trickles and trickles back down into the boiling flask. The extractions were carried out at 60, 65, 70, 75 and 80°C extraction temperatures, ϵ_{tp} and 60, 90, 120, 150 and 180 min extraction time, ϵ_{tm} respectively. The flask containing the mixture of solvent and the extracted oil was removed at the end of the extraction process and placed in the water bath and heated at 80°C for hours to evaporate the solvent from the extracted oil, which was weighed and taken as a fraction of the ground seed samples. All experiments were replicated trice. The oil yield was calculated from Equation 3 as adopted by [14] for determination of percentage oil yield.

$$\text{Oil Yield (\%)} = \frac{\text{Wiegth of oil extracted}}{\text{Wiegth of sandbox seed sample used in the extraction}} \times 100 \quad (1)$$

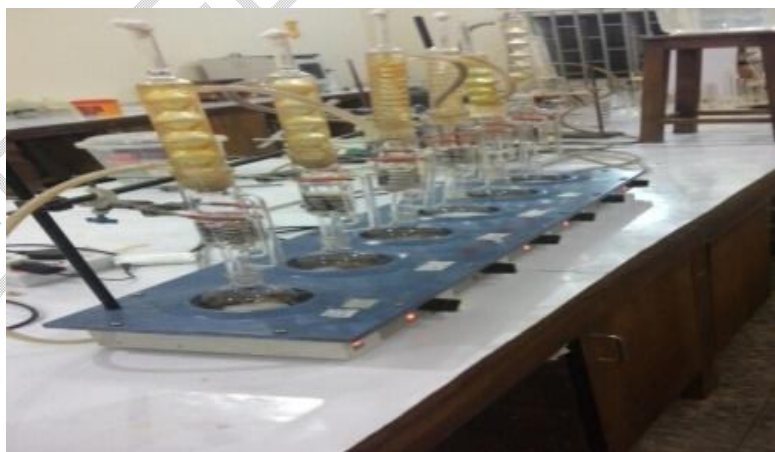


Fig. 2. Soxhlet Apparatus

Response Surface Methodology (RSM)

RSM Design Expert software package (6.0.6) was used to design the experiment. Keying in the ranges of process variables into the design expert, produced sets of experimental factor combinations used for the experiment. Oil extracted from each of the runs were keyed into the software was keyed in as the response of the particular combination. The models that

made up the Design Expert includes linear, the two factorial interactions (2FI), the quadratic and the cubic models respectively. These models utilize the probability of error value (p-value) and coefficient of determination (R^2) to analyze the outcomes. These two statistical parameters interpret level of relationship between process factors and oil yield. Thus, the choice of the best model for the oil extraction was based on values of their p and R^2 respectively. The selected model was analyzed using Analysis Of Variance (ANOVA) to further ascertain the model's level of significance and fitness in explaining the relationship between the process factors and oil yield. Windows 20.0 SPSS statistical software package was used to analyze the tests of between-subjects of effects of processing conditions on oil yield. The sets of process parameter combination suggested by the model to be optimal for the oil extraction were used to conduct a validation experiment. Finally, the experimental results were compared with predicted values for similarities.

3. RESULTS AND DISCUSSION

The results of the oil yield at various process conditions are presented in Table 1, while charts relating the effects of process parameters on oil yield are shown in Figs 3-6. The oil yield by solvent extraction ranged from 20.86-53.60. In comparison to oil yields from sandbox seed by solvent extraction, the highest oil yield of 53.60% was similar to 53.61 and 53.81% obtained by [32] and [29], and higher than 37.75, 36.70 and 42.70% obtained by [4],[38] and [31]. The value was however lower than 57.26% obtained by [37]. According to [8, 35], differences in oil yield during extraction is a function of extraction methods employed, and also biological and environmental conditions.

Table 1. Oil Yield from Sandbox Seed at Various Processing Conditions

Run	Factor 1 A: mc (%)	Factor 2 B: τ_{tp} (%)	Factor 3 C: τ_{tm} (min)	Factor 4 D: ε_{tp} (°C)	Factor 5 E: ε_{tm} (h)	Response 1 Oil yield (%)
1	8	90	10	70	2.0	45.76
2	8	90	10	70	3.0	35.02
3	8	90	10	70	1.0	23.00
4	6	85	5	65	2.5	38.33
5	8	90	10	60	2.0	42.87
6	10	85	15	75	1.5	40.00
7	10	85	5	75	2.5	40.12
8	4	90	10	70	2.0	20.86
9	10	85	15	65	2.5	48.88
10	8	90	10	80	2.0	44.00
11	12	90	10	70	2.0	30.00
12	8	90	10	70	2.0	45.00
13	6	95	15	65	2.5	40.00
14	8	90	10	70	2.0	50.00
15	8	90	10	70	2.0	49.80
16	10	95	15	75	2.5	46.54
17	6	85	15	65	1.5	35.66
18	10	95	5	65	2.5	36.00

19	8	80	10	70	2.0	38.10
20	8	90	10	70	2.0	47.77
21	10	85	5	65	1.5	36.56
22	6	95	15	75	1.5	39.00
23	10	95	5	75	1.5	42.40
24	6	85	5	75	1.5	39.60
25	6	85	15	75	2.5	40.55
26	6	95	5	65	1.5	40.06
27	6	95	5	75	2.5	38.99
28	8	90	10	70	2.0	46.44
29	8	90	0	70	2.0	21.00
30	10	95	15	65	1.5	53.60
31	8	90	20	70	2.0	47.99
32	8	100	10	70	2.0	51.33

Where mc = moisture content of sandbox seed, r_{tp} = Roasting temperature, r_{tm} = Roasting time, ϵ_{tp} = Extraction temperature and ϵ_{tm} = Extraction time

Oil yield increased with increase in the seed moisture content from 4% up to 10%, after which a decrease in the oil yield was observed (Fig. 3 and 4). The highest oil yield was obtained at 10% seed moisture. The decrease in sandbox oil yield at moisture above 10% could be attributed to the observation that at moisture content above 10%, ground sandbox seed forming a slurry paste which made it somewhat difficult for the extraction solvent (petroleum ether) to pick oil as it passes through the sample during refluxing process. The trend is in agreement with reports on soybeans, un-sieved and sieved rice bran [26, 39] among others.

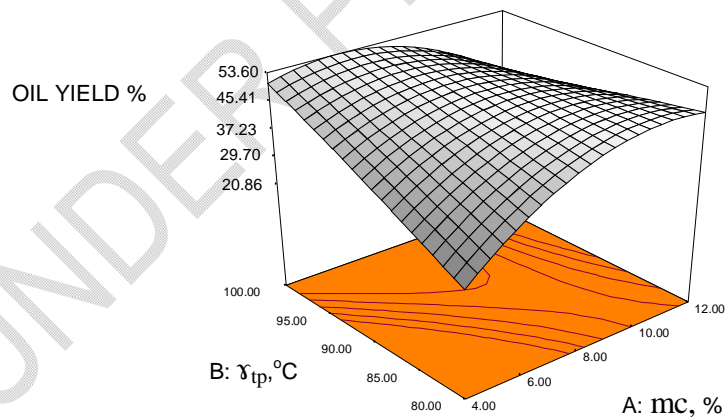


Fig. 3. Oil yield against roasting time and moisture content

Oil yield increased with increase in roasting temperature. The highest oil yield occurred at the roasting temperature region of 95-100°C (Fig. 4). This might be due to the fact that sandbox seed is a soft seed, very similar to melon seed that is easily crushed with the finger. The seed required mild roasting below 100°C as such temperature is enough to penetrate

the seed to release oil from oil bearing cells. Similarly, 100°C roasting temperature was obtained for maximum oil yield of from *Moringaoleifera*[2].

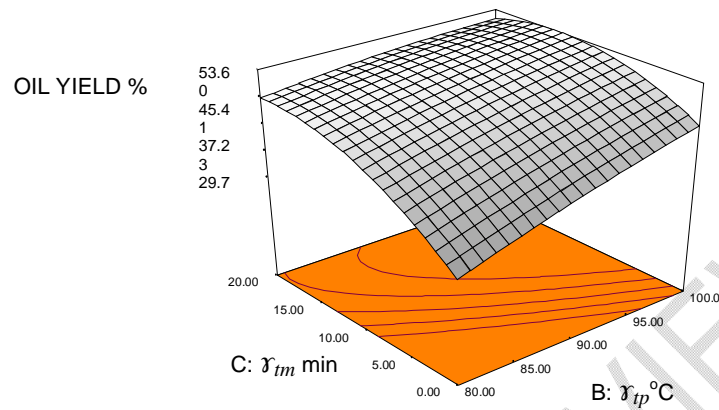


Fig. 4. Roasting time and reaction temperature against oil yield

The roasting temperature was however lower than 130°C and 150°C obtained by [6] and [18] for maximum oil yield from palm kernel and sesame seed respectively.

Oil yield was lowest for the unroasted samples at 0 min roasting time and increased with increase in roasting time up to 15 min beyond which it linearized (Fig. 4).

The oil yield increased with increase in extraction temperature from 60°C up to 75°C, after which the oil yield decreased (Fig. 5). Extraction temperature for maximum oil yield from seeds and nuts by solvent extraction have been found to range between 60-90°C. 60°C extraction temperature was found to be as adequate for maximum oil extraction from *Jatropha curcas* L. kernel[9]. However, [41], reported maximum oil extraction from bitter melon at 98°C extraction temperature and [18] reported a lower extraction temperature of 40°C for maximum oil yield from sesame seed.

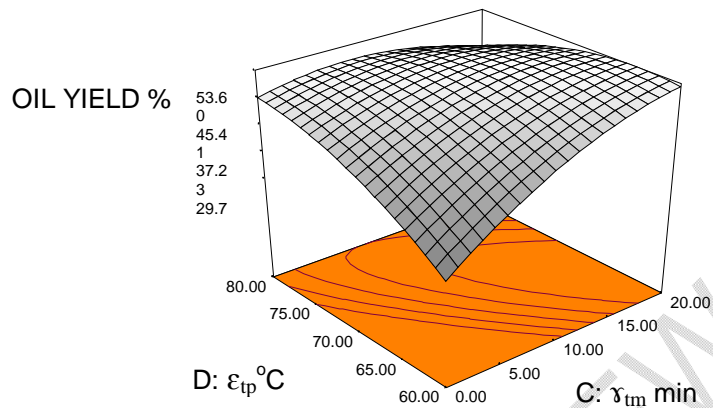


Fig. 5 Extraction time and roasting temperature against oil yield

Oil yield increased with increase in extraction time from 60 min up to 120 min, beyond which a decrease in oil yield was observed. The trend from these graphs suggests that 2 hours reaction time is adequate to completely remove oil from sandbox seed by solvent extraction. The findings are analogous to the ones observed by [41], who obtained a maximum oil yield from bitter melon at 90 min. [26] obtained maximum oil yield from soybeans at extraction time of 2½ hour, while below and above this time the oil yield reduced, and [18] obtained maximum oil yield from sesame seed at 1 hour extraction time.

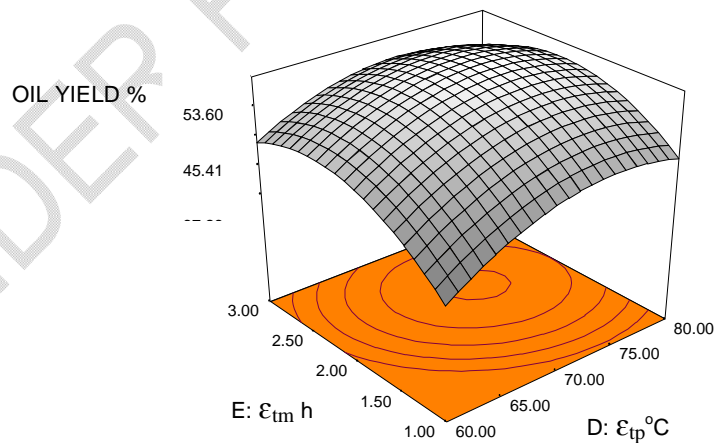


Fig. 6.Extraction time and temperature against oil yield

RSM optimization of oil extraction from sandbox seed

Statistical parameters such as the polynomial order with the largest number non- aliasing significant additional terms, insignificant lack-of-fit and high Adjusted and Predicted (R^2) were

considered in selecting the best model to predict the oil yield from the sandbox seed. From the four different models, the quadratic model with the highest R^2 and lower standard deviation values (Table 2) was selected.

Table 2. Model Comparison

Statistics	Models			
	Linear	2Factorial Interaction	Quadratic	Cubic
Standard Deviation, SD	4.89	5.91	3.77	2.26
R^2	0.5663	0.6083	0.8127	0.9089
Mean	29.39	29.39	29.39	29.39
Adjusted R^2	0.4789	0.2853	0.7021	0.8919
Coefficient of Variation, C.V.	16.49	19.95	12.69	7.55
Predicted R^2	0.3920	-2.1384	-1.8179	-19.9677
PRESS	847.75	4352.16	3906.39	29140.86
Adequate Precision	9.275	5.467	7.288	10.360

Where PRESS = Predicted Sum of Square.

Model equation for predicting oil yield from sandbox seed relative to the process factors is given in Eq. 5.

$$\begin{aligned}
 OY = & 45.89 + 2.09mc + 1.81r_{tp} + 3.59r_{tm} + 0.013\epsilon_{tp} + 1.10\epsilon_{tm} - 1.91mc^2 - 0.88r_{tp}^2 - \\
 & 1.67r_{tm}^2 + 0.56\epsilon_{tp}^2 - 3.05\epsilon_{tm}^2 + 0.57mc r_{tp} + 2.23mc r_{tm} - 0.63mc\epsilon_{tp} - 0.29mc\epsilon_{tm} + 0.70r_{tp}r_{tm} \\
 & - 0.23r_{tp}\epsilon_{tp} - 1.85r_{tp}\epsilon_{tm} - 1.39r_{tm}\epsilon_{tp} + 0.80r_{tm}\epsilon_{tm} + 0.50\epsilon_{tp}\epsilon_{tm} \quad (5)
 \end{aligned}$$

Where, OY = Oil Yield (%), mc = moisture content of sandbox seed, r_{tp} = Roasting temperature, r_{tm} = Roasting time, ϵ_{tp} = Extraction temperature and ϵ_{tm} = Extraction time [Std. Dev. = 3.77, R^2 = 0.8127, Mean = 29.39, Adj R^2 = 0.7021, C.V. = 12.69, Pred. R^2 = -1.8179, PRESS = 3906.39, Adeq. Precision = 7.288 and F-value = 4.50 (Tables 2 and 3)]

As shown in the equation, positive signs indicate that oil yield varies directly with the factors while negative sign indicates an inverse relationship amongst the factors. The values of "Prob> F" in Fig. 3, lower than 0.05, such as r_{tm} , ϵ_{tp} , ϵ_{tm} , mc^2 , r_{tm}^2 , ϵ_{tp}^2 , and ϵ_{tm}^2 , represents significant model parameters for sand box oil extraction.

Table 3. ANOVA for Response Surface Quadratic Model of the Oil Extraction

Source	Sum of squares	DF	Mean square	F value	Prob > F
Model	1442.72	20	72.14	4.50	0.0059 ^s
mc	68.56	1	68.56	4.21	0.0142
r_{tp}	0.052	1	0.052	0.0016	0.9688
r_{tm}	181.60	1	181.60	10.97	0.0065 ^s
ϵ_{tp}	50.82	1	50.82	0.42	0.0086 ^s
ϵ_{tm}	668.10	1	668.10	41.06	0.0001 ^s

mc^2	116.76	1	116.76	7.47	0.0140 ^s
γ_{tp}^2	0.33	1	0.33	0.016	0.9026
γ_{tm}^2	101.43	1	101.43	7.47	0.0174 ^s
ϵ_{tp}^2	151.01	1	151.01	12.36	0.0058 ^s
ϵ_{tm}^2	98.20	1	98.20	7.11	0.0220 ^s
$mc\gamma_{tp}$	2.44	1	2.44	0.18	0.6825
$mc\gamma_{tm}$	0.16	1	0.16	0.011	0.9179
$mc\epsilon_{tp}$	20.86	1	20.86	1.36	0.2677
$mc\epsilon_{tm}$	7.65	1	7.65	0.47	0.5084
$\gamma_{tp}\gamma_{tm}$	4.14	1	4.14	0.082	0.7795
$\gamma_{tp}\epsilon_{tp}$	1.054	1	1.054	0.004	0.9513
$\gamma_{tp}\epsilon_{tm}$	5.51	1	5.51	0.25	0.6246
$\gamma_{tm}\epsilon_{tp}$	19.28	1	19.28	1.25	0.2875
$\gamma_{tm}\epsilon_{tm}$	8.69	1	8.69	0.63	0.4449
$\epsilon_{tp}\epsilon_{tm}$	1.47	1	1.47	0.11	0.7506
Residue	152.21	11	13.84		
Lack of fit	149.68	6	24.95	49.25	0.0003 ^s
Pure Error	2.53	5	0.51		
Cor Total	1393.74	31			

The quadratic model with R^2 of .81 and p-value < .0001 (Table 4) was concluded to be significant and thus selected. The R^2 of .81 is an indication of strong correlation between the sandbox oil yield and the process factors. This represents the degree of confidence that the model explained 81.27% of every irregularity as regards the process factors and oil yield.

Table 4. Test of between-subjects effect of process conditions on oil yield from sandbox seed

Source	Df	Mean Square	F	Significance
Corrected Model	27	51.439	86.003	0.0001 ^s
Intercept	1	10656.051	17816.504	0.0001 ^s
mc	3	67.567	112.970	0.0001 ^s
γ_{tp}	2	4.189	7.004	0.049
γ_{tm}	2	122.883	205.456	0.0001 ^s

ϵ_{tp}	2	124.977	208.957	0.0001 ^s
ϵ_{tm}	2	164.746	275.449	0.0001 ^s
Error	4	0.598		
Total	32			
Corrected Total	31			

The model F-value of 4.50 (Table 3) is an indication of how well the model explained the correlation between process factors and oil yield. The quadratic curve depicts optimum and minimum points. That indicates that there are values of process parameters points when oil yield would be minimum or maximum.

Model validation

A similarity plot of correlation relationship of the laboratory results and predicted results of oil yield from the sandbox seed is shown in Fig. 7. The R^2 and p values means that eliminating errors resulting from real experiments, that the model can reliably estimate extractable oil from sandbox seed by solvent means within the range of process variables studied. At the range of process factors: 8-12% wb moisture content, 80-100 °C roasting temperature, 0-20 min roasting time, 60-80°C extraction temperature and 60-180 min extraction time.

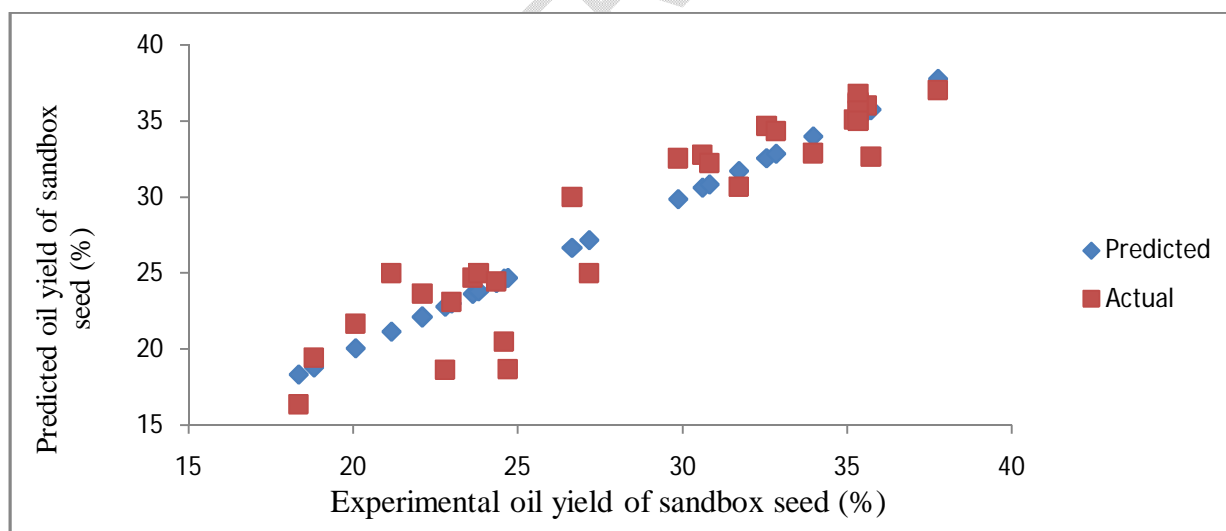


Fig. 7. Predicted oil yield against actual oil yield

The maximum oil yield of 53.60% was obtained at 10% wb moisture content, 95°C roasting temperature, 15 min roasting time, extraction temperature of 65°C and 90 min extraction time, while the predicted optimum oil yield was 57.10% at processing conditions of 10.25% wb moisture content, 98.72°C roasting temperature, 17.63 min roasting time, extraction temperature of 67.10°C and 96.6 min extraction time. The oil yield from the experiment carried out using the predicted optimum conditions was 56.89%. This value validated the

predicted oil yield and processing conditions. The differences between the results of the actual experiment and predicted values were very low, ranged from 0.01- 0.60. Indicating how well the model predicted the oil yield from sandbox seed by solvent method.

4. CONCLUSION

Oil extraction process from sandbox seed by solvent method was optimized. From the range of process variables evaluated, the sandbox seed oil yield varied from 20.86-53.60. The maximum oil yield of 53.60% was obtained when the seed samples were subjected to 10% wb moisture content, 95°C roasting temperature, 15 min roasting time, extraction temperature of 65°C and 90 min extraction time. The predicted maximum oil yield was 57.10%, corresponding to processing conditions of 10.25% wb moisture content, 98.72°C roasting temperature, 17.63 min roasting time, extraction temperature of 67.10°C and 96.6 min extraction time. The oil yield from the experiment carried out using the predicted optimum conditions was 56.89%. The differences between the results of the actual experiment and predicted values were very low, ranged from 0.01-0.60. The R^2 of 0.81 for model developed for the sandbox seed oil extraction was an indication of high correlation between the process factors and oil yield. The high parity between the results of actual experiments and predicted values showed that the model sufficiently predicted the optimization process.

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