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STATISTICAL MODELLING OF OIL EXPRESSION FROM NEEM SEED USING A SCREW PRESS

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Abstract
A study was carried out to determine the effect of some process parameters on the yield of neem seed oil from a screw press using 4^4 factorial design experiments. This consisted of four independent, randomly assigned parameters at four levels each namely, moisture content (wb) of seeds (6.3%; 8.1%, 13.2% and 16.6%); temperature of heating at Θ_1 (55°C), Θ_2 (70°C), Θ_3(85°C) and Θ_4 (100°C); duration of heating at t_1 (5 min.), t_2(10 min.), t_3 (15 min.) and t_4 (20 min.) and machine pressure at p_1 (602.91 kN/m^2), p_2 (723.07 kN/m^2), p_3(925.84 kN/m^2) and p_4(1334.88 kN/m^2). Three replicates of the experiments were carried out and from the values obtained, oil yield was calculated. The effects of the processing conditions (moisture content, heating duration, heating temperature and machine pressure) on the oil yield were investigated using analysis of variance (ANOVA) at $p \leq 0.05$ and the levels of the significant means were further evaluated using Duncan's New Multiple Range Test (DNMRT). Mathematical model was developed to express the oil yield with respect to the process parametersand this was fitted to experimental data using MATLAB 8.0 software package. Adequacy of the model was authenticated using coefficient of determinationand adjusted coefficient of determination, R^2.The results obtained showed that temperature, heating time, moisture content, pressure and their interactions all proved to determine the outcome of oil yield of neem seed. The highest oil yield of 33.55% was obtained at temperature of 81.98°C, pressure of 1067.23kN/m^2, moisture content of 9.03% and heating duration of about 20 min.Theresults obtained(coefficient of determination and adjusted coefficient of determination R^2) indicated that the model generated was statistically adequate.

Keywords: Neem seed, Oil, Factorial design, Moisture content, Temperature

1. Introduction

The neem (*Azidirachtaindica*) tree is popularly known as *dongoyaro* in the Northern part of Nigeria where it grows in abundance. All parts of the tree have been reported to be very useful (Adewoye and Ogunleye, 2012). They also reported that the most famous product of the tree is the oil obtained from the seed kernel. Neem seed kernel is a component of the neem fruit which has high concentration of oil (Ikasari and Indraswati, 2008), Ahmed and Grainge (1985) reported that neem seed contains 35 – 45% oil. The quality of the oil differs according to the method of processing.

Oil extraction is the process of expelling oil from oil bearing agricultural seeds and there are different methods employed in the extraction process. These include the traditional method, supercritical fluid extraction method, mechanical method and the solvent extraction process (contact equilibrium process) or a combination of mechanical and solvent extraction processes (Oyinlola and Adekoya, 2004). Oil extraction by mechanical pressing is simpler, safer and contains fewer steps, compared to oil extraction by solvent (Oyinlola and Adekoya, 2004). Mechanical expression of oil involves the application of pressure (using hydraulic or screw presses) to force oil out of the oil bearing material.

Heat treatment of oil seeds has been observed to rupture the oil bearing cells of the seed, coagulate the protein in the meal, adjust the moisture level of the meal to optimum level for oil expression, lower the viscosity and increase the fluidity of the oil to be expelled, thereby facilitating oil expression from the material (Adeeko and Ajibola, 1990). Effect of different processing factors on yield and quality of oil from some oil bearing seeds have also been investigated by various authors. These include groundnut (Adeeko and Ajibola, 1990); olive (Torres and Maestri, 2006); sesame seed (Akinoso *et al*., 2006a), palm kernel (Akinoso, 2006); soyabean (Tunde-Akintunde *et al.*, 2001); conophor nut (Fasina and Ajibola, 1989), castor seed (Shridhar *et al*., 2010), and neem seed (Adewoye and Ogunleye, 2012). Adewoye and Ogunleye (2012) used the response surface methodology to optimize oil extraction from neem seed.

Neem oil is used as a base for variety of organic cosmetics including soaps, shampoos, hand and body lotions and creams (Rajev, 2009). It is also used as an organic bio-pesticide repellant against insects such as meal worms and aphids (Rajev, 2009). Abdullahi (2004) reported thatneem oil is used for treating many skin diseases such as eczema, psoriasis (skin disease) and skin allergies.
Modelling reduces number of experiments; thereby reducing time and expenses and providing process optimization, predictive capability, improved process automation and control possibilities (Sablani *et al.* 2006) (The yield of the oil obtained by mechanical expression are affected by various operating conditions such as heating temperature, heating time, moisture content, applied pressure, particle size and pressing time (Khan and Hanna, 1983). According to Bamgboye and Adejumo (2011), for maximum oil yield and least residual oil in cake, it is very important to control these conditions during the extraction process. Thus, for efficient mechanical expression, a careful establishment of optimum processing conditions is necessary) The intention of this study therefore is to determine the effect of moisture content, temperature of heating, duration of heating and pressure on the yield of neem seed kernel oil expressed using an oil expeller and to develop model equations to optimise oil expression from neem seed kernels.

2.0 Materials and Methods

2.1 Materials

Mature and healthy neem seeds used for the experiments were obtained from Katsina Zonal Forest Office, Ministry of Agriculture and Natural Resources, Katsina, Katsina State, Nigeria. A neem seed roaster which uses steam as a source of heat energy was used in heating the neem seeds prior to oil expression. NCRI, Badeggi developed oil expeller was used in carrying out oil expression. The expeller capacity ranged from 15 – 20 kg/h and was powered by a 7.5 kW, 3 phase electric motor with in-built reduction gear. It was run at 75 rpm.

Fig 1: NCRI, Badeggi Developed Oil Expeller

2.2 Methods

The following procedures were used in carrying out the experiments.

2.2.1 Material Preparation

The seeds still within their endocarp were sun-dried for two days to allow for their easy removal. The dried endocarp was cracked to obtain the seed kernel and after decortication, the hulls of the seeds and other contaminants were removed by winnowing.

2.2.2 Moisture Content Determination

The moisture content of the seeds was determined using ASABE (2008) standard for oil seeds. Three samples each weighing 15 g were placed in an oven set at 105°C and dried for 24 hours. The samples were then cooled, weighed and the moisture content calculated. Loss in weight was assumed to be moisture loss. Initial moisture content of the seeds was 8.1%. The sample was divided into four parts; one part (one-fourth) was left as it was; while the remaining parts were sun-dried at 34°C for 12 hours to further reduce the moisture content to 6.3%. This sample was further divided into three parts, one part (one-third) was left as it was; while the remaining parts were further conditioned to desired moisture content levels as described by Akinoso (2006). Adding distilled water as calculated from equation 1 increased the moisture content of the seeds according to Akinoso (2006)

$$Q = \frac{A(b-a)}{(100-b)} \tag{1}$$

where:

A = Initial mass of the sample

a = Initial moisture content of the sample, % wb

b = Final (desired) moisture content of sample % wb

Q = Mass of water to be added kg

Each sample was sealed in a separate polythene film. The samples were kept at 5°C in a refrigerator for a week to enable the moisture to distribute uniformly throughout the samples. The four moisture content levels that were prepared are: 6.3% wb, 8.1% wb, 13.2% wb and 16.6% wb. Based on literature search, I decided to go below and slightly above the values seen.

2.2.3 Heating of Seeds

A seed roaster of 15kg/hr capacity, which uses steam as a source of heat energy was used in heating the neem seeds prior to expelling of oil from them. The already conditioned seeds were poured in the fabricated seed roaster which had been turned on before now. As soon as the required temperature was reached, the samples were poured inside for the required heating duration. A thermometer (which measures the temperature of the seeds being heated) was attached to the seed roaster. Neem kernel samples of 2 kg each were heated in the seed roaster at different temperature and time combinations. A stopwatch was used to monitor the time. As soon as the specified heating duration was reached, the seeds were discharged/released from the roaster through its seed discharge outlet directly into the hopper of the oil expeller.

2.2.4 Determination of Machine Pressure

Preliminary testing was done to determine (calculate) the pressure generated within the system: the diameter of the barrel of the oil expeller was a constant and this was known; worm was loosened completely, the worm shaft had four different points with different diameters (measured, noted and marked), these points on the worm correspond to other points on the shank (outer part of the worm shaft which could be seen even when the worm shaft is inside the barrel) which were already marked, these points on the shank could be seen as the shaft was adjusted. The more the worm shaft was adjusted inwards through the adjustment mechanism, the higher the diameter covered by the worm and the lesser the clearance between the worm and the barrel and thus the more the pressure generated within the system. The measured diameters at the different points on the worm shaft are 0.07, 0.072, 0.074 and 0.076m. These values were substituted into the formula reported by Hannah and Stephens (1984) to obtain the Torque (equation 2).

$$P_W = \frac{2\pi NT}{60} \qquad (2)$$

Where:
P_W = Power, Watts
N= Revolutions per minute
T= Torque, Nm

$$\text{Torque} = \text{Force} \times \text{Distance} \qquad (3)$$

The torque and the diameters calculated were then substituted into equation 2 to get the different pressures exerted by the machine.
Also,

$$P_r = \frac{F}{A} \qquad (4)$$

Where:
P_r= Pressure, N/m^2
F = Force, N
A= Area, m^2
The applied pressures calculated were 602.91, 723.07, 925.84 and 1,334.88 kN/m^2, respectively

2.2.5 Oil Expression

Neem seed oil was expressed using NCRI, Badeggi multi-seed screw press (Fig.1).The expeller capacity ranged from 15–20 kg/h and was powered by a 7.5 kW, 3 phase electric motor with in-built reduction gear. It was run at 75 rpm.

The experimental procedure was carried out after running the screw press for about 3 min before loading the pre-treated samples as described by Akinoso (2006). Oil expressions were conducted at moisture contents of 6.3, 8.1, 13.2 and 16.6% wb. They were heated at 55, 70, 85 and 100°C at 5, 10, 15 and 20 min heating duration using a steam roaster. The applied pressure was 602.91, 723.07, 925.84 and 1,334.88kN/m^2, respectively. Oil expressed and the cakes from the samples were collected separately. Cleaning of the expeller barrel was done after each expression. The experiments were replicated three times. The data obtained for oil yield was regressed using multiple regression analyses in MATLAB (8.0) computer software.

2.2.6 Determination of Oil Yield

The expressed oil was collected and left to stand for 96 hours as recommended by Olajide (2000) so that the oil can be clarified and the volume measured. The weights of the cakes were determined using an electronic weighing balance. The recorded oil yield was compared with the initial oil content of the seed.

Soxhlet oil extraction method as reported by Akinoso*et al.* (2006a) was applied to determine the initial oil content of the seeds. The mean value of three samples was expressed as percentage content as follows:

$$\%\,Oil\ Content = \frac{Weight of oil in Solution}{Weight of seed samples} \times 100 \tag{5}$$

The weights of the oils expressed were obtained using a weighing balance. Percentage oil yield and Expression efficiency were calculated using equations (1) and equation (2) respectively.

$$\%\,OilYield = \frac{Weight of oil}{Weight of expressed seed} \times 100 \tag{6}$$

$$\%\,ExpressionEfficiency = \frac{OilYield}{TotalOilContent} \times 100 \tag{7}$$

2.3 Statistical Analysis

A 4^4 full factorial design was used for the experimentation. Four parameters were measured at four levels each which gave 256 treatment combinations. These parameters were moisture content of neem seeds, machine pressure, duration and temperature of heating; and these were independent variables while oil yield was the dependent variable. The parameters and their levels are shown in Tables 1. The choice of moisture content of seeds, machine pressure, duration and temperature of heating were influenced from literature.

The necessary parameters were varied taking into consideration the reported values for oil seeds. Obtained data from these experiments formed the basis for the model equation. The parameters m_1, m_2, m_3, m_4 are moisture contents at 6.3, 8.1, 13.2 and 16.6 %, respectively; Θ_1, Θ_2, Θ_3, Θ_4 are temperatures at 55, 70, 85 and 100°C, respectively; p_1, p_2, p_3, p_4 are machine pressures at 602.834, 743.628, 978.628 and 1,449.12kN/m^2, respectively while t_1, t_2, t_3, t_4 are heating durations at 5, 10, 15, and 20 minutes, respectively (Table 1).All the treatments were randomly assigned.

Table 1: Parameters and their Levels

Parameters	Levels			
	1	2	3	4
Moisture Content % wb , m	6.3	8.1	13.2	16.6
Heating Temperature $^{\circ}$C, Ѳ	55	70	85	100
Heating Duration (min), t	5	10	15	20
Machine Pressure (KN/m^2), p	602.91	723.07	925.84	1334.88

The effect of the processing conditions (moisture content, heating temperature, heating duration and machine pressure) on yield of the oil was investigated using analysis of variance (ANOVA) at $p \leq 0.05$ and levels of the significant means were further evaluated using Duncan's New Multiple Range Test (DMRT).

The data was analysed using multiple regression technique of the MATLAB (8.0) software to generate a mathematical model. Adequacy of the model generated was authenticated by the coefficient of determination and adjusted coefficient of determination R^2.

2.4 Model Development

All the possible relationships between the process conditions manipulated (temperature, pressure, moisture content and heating duration) and the measured output (oil yield) were explored with a view to selecting models that appropriately capture the relationship between the input and output

parameters. The model development process therefore took cognizance of the various relationships exhibited between the process conditions and the output.
The data obtained for oil yield was regressed using multiple regression analyses in MATLAB (8.0) computer software. The essence is to find functional relationship that can adequately relate process parameters (temperature, pressure, heating duration and moisture content) to the measured output (oil yield) Boonmee *et al.* (2010) reported that multiple regression analysis is used as tools of assessment of the effects of two or more independent factors on the dependent variables.

From the regression analysis carried out, the best performing functional model was developed. The criteria for adjudging this model was the value of the coefficient of determination and adjusted coefficient of determination. Models were checked for adequacy using these statistics and the one found to be adequate was selected from among the other possible combinations of the models. The coefficients of determination R^2 is a measure of the total variation of the observed values of the extracted oil about the mean explained by the fitted model (Shridhar *et al*, 2010).

3.0 Results and Discussion

3.1 Moisture Content of the Neem Seeds

The mean initial moisture content of the neem seeds based on triplicate determinations was 8.1%. This was further reconditioned to 6.3, 13.2 and 16.6%.

3.2 Initial Oil Content of Neem Seeds prior to Treatment

The average initial oil content of the seeds was 37.71%

3.3 Percentage Oil Yield of Treated Neem Seeds

Oil yield of the different treatment combinations was calculated and shown in Table 3.

Table 3: Results of Oil Yield

S/No	Treatment combination	Oil Yield		
		Replicate 1	Replicate 2	Replicate 3
1	m1t1p1β1	13.47	12.98	13.42
2	m1t1p1β2	13.35	13.37	13.34
3	m1t1p1β3	13.56	13.42	12.92
4	m1t1p1β4	13.72	13.55	12.96
5	m1t1p2β1	16.07	15.92	15.95
6	m1t1p2β2	15.98	15.94	16.03
7	m1t1p2β3	16.17	16.12	15.97
8	m1t1p2β4	16.21	16.02	16.29
9	m1t1p3β1	20.21	20.33	20.17
10	m1t1p3β2	20.72	20.67	20.83
11	m1t1p3β3	21.77	20.89	21.71
12	m1t1p3β4	22.55	21.97	22.32
13	m1t1p4β1	18.21	18.17	18.09
14	m1t1p4β2	19.01	18.67	18.98
15	m1t1p4β3	19.16	18.93	19.12
16	m1t1p4β4	20.47	19.87	20.22
17	m1t2p1β1	15.72	15.86	16.01
18	m1t2p1β2	17.12	16.97	17.09

19	m1t2p1β3	17.17	17.19	16.99
20	m1t2p1β4	17.22	17.37	17.09
21	m1t2p2β1	19.99	19.11	19.22
22	m1t2p2β2	20.02	20.61	20.99
23	m1t2p2β3	22.17	21.78	21.54
24	m1t2p2β4	21.82	22.35	22.77
25	m1t2p3β1	25.21	24.72	24.01
26	m1t2p3β2	26.77	25.91	26.74
27	m1t2p3β3	27.06	28.92	27.22
28	m1t2p3β4	29.74	29.61	29.42
29	m1t2p4β1	22.78	21.87	22.92
30	m1t2p4β2	23.11	23.09	23.21
31	m1t2p4β3	24.27	24.17	24.28
32	m1t2p4β4	24.98	23.44	24.67
33	m1t3p1β1	18.32	18.17	18.38
34	m1t3p1β2	20.11	19.96	20.28
35	m1t3p1β3	22.02	21.89	22.11
36	m1t3p1β4	23.13	23.06	23.15
37	m1t3p2β1	25.07	25.12	24.93
38	m1t3p2β2	26.82	27.04	26.63
39	m1t3p2β3	27.97	27.92	28.04
40	m1t3p2β4	28.53	28.64	28.43
41	m1t3p3β1	30.07	29.97	30.09
42	m1t3p3β2	31.81	31.72	31.95
43	m1t3p3β3	32.99	32.78	32.86
44	m1t3p3β4	34.17	33.92	34.09
45	m1t3p4β1	28.87	28.65	28.93
46	m1t3p4β2	29.35	29.41	29.22
47	m1t3p4β3	31.22	31.28	30.96
48	m1t3p4β4	31.72	31.59	31.52
49	m1t4p1β1	18.12	18.23	17.97
50	m1t4p1β2	18.17	18.09	18.24
51	m1t4p1β3	17.37	16.82	17.17
52	m1t4p1β4	17.99	18.04	17.87
53	m1t4p2β1	21.08	21.11	21.99
54	m1t4p2β2	21.97	21.72	21.54
55	m1t4p2β3	21.14	21.52	20.92
56	m1t4p2β4	21.32	21.41	21.96
57	m1t4p3β1	25.32	25.11	25.54
58	m1t4p3β2	24.87	25.22	24.98
59	m1t4p3β3	26.11	25.84	26.03

60	m1t4p3β4	26.20	26.12	26.29
61	m1t4p4β1	24.21	24.04	24.33
62	m1t4p4β2	23.98	23.27	23.72
63	m1t4p4β3	24.02	24.94	24.14
64	m1t4p4β4	25.22	26.13	25.18
65	m2t1p1β1	15.34	15.12	14.29
66	m2t1p1β2	16.21	16.01	15.72
67	m2t1p1β3	15.77	15.56	15.32
68	m2t1p1β4	16.01	16.54	15.82
69	m2t1p2β1	18.35	19.03	18.66
70	m2t1p2β2	20.98	21.04	20.75
71	m2t1p2β3	22.77	22.13	21.94
72	m2t1p2β4	23.96	23.07	24.15
73	m2t1p3β1	22.11	23.03	23.46
74	m2t1p3β2	23.72	23.92	23.73
76	m2t1p3β4	27.27	26.83	26.51
77	m2t1p4β1	20.16	21.72	20.91
78	m2t1p4β2	22.32	21.92	22.41
79	m2t1p4β3	24.97	23.18	23.24
80	m2t1p4β4	25.02	25.78	26.11
81	m2t2p1β1	18.33	17.62	17.97
82	m2t2p1β2	20.11	19.97	19.82
83	m2t2p1β3	20.79	21.83	22.92
84	m2t2p1β4	21.55	23.10	22.99
85	m2t2p2β1	23.65	23.33	23.72
86	m2t2p2β2	25.86	25.22	25.19
87	m2t2p2β3	27.09	26.84	26.53
88	m2t2p2β4	28.12	27.55	28.05
89	m2t2p3β1	28.88	29.21	28.83
90	m2t2p3β2	29.91	29.07	30.76
91	m2t2p3β3	30.74	29.83	30.62
92	m2t2p3β4	31.41	32.03	31.74
93	m2t2p4β1	25.23	26.08	25.17
94	m2t2p4β2	26.45	26.59	27.21
95	m2t2p4β3	26.07	25.67	27.08
96	m2t2p4β4	25.99	26.34	26.46
97	m2t3p1β1	20.27	19.86	20.52
98	m2t3p1β2	22.08	22.73	22.95
99	m2t3p1β3	22.98	23.51	23.83
100	m2t3p1β4	23.19	23.94	23.57
101	m2t3p2β1	26.63	25.83	26.22

102	m2t3p2β2	28.92	28.76	28.51
103	m2t3p2β3	30.92	31.08	29.43
104	m2t3p2β4	31.18	31.93	31.67
105	m2t3p3β1	32.77	33.12	32.19
106	m2t3p3β2	32.99	34.02	33.73
107	m2t3p3β3	34.28	35.11	34.02
108	m2t3p3β4	36.12	36.72	36.23
109	m2t3p4β1	28.11	28.93	28.72
110	m2t3p4β2	30.82	29.47	30.68
111	m2t3p4β3	32.98	31.33	31.54
112	m2t3p4β4	33.44	32.97	33.08
113	m2t4p1β1	19.22	19.17	19.11
114	m2t4p1β2	18.43	18.22	18.17
115	m2t4p1β3	17.88	18.13	17.54
116	m2t4p1β4	18.52	18.03	18.21
117	m2t4p2β1	21.17	22.02	21.34
118	m2t4p2β2	22.43	21.92	22.21
119	m2t4p2β3	22.98	21.63	22.42
120	m2t4p2β4	22.22	21.17	23.04
121	m2t4p3β1	26.82	26.11	26.49
122	m2t4p3β2	26.17	25.23	25.77
123	m2t4p3β3	27.01	26.15	27.22
124	m2t4p3β4	27.21	27.53	27.33
125	m2t4p4β1	25.26	25.72	24.98
126	m2t4p4β2	25.73	25.46	25.61
127	m2t4p4β3	26.12	25.34	26.07
128	m2t4p4β4	26.26	26.72	25.41
129	m3t1p1β1	12.71	12.32	12.46
130	m3t1p1β2	12.98	13.14	12.73
131	m3t1p1β3	13.33	13.49	13.22
132	m3t1p1β4	13.16	13.32	13.58
133	m3t1p2β1	16.23	16.17	16.62
134	m3t1p2β2	16.79	17.14	17.24
135	m3t1p2β3	17.92	17.63	17.51
136	m3t1p2β4	17.77	18.21	18.05
137	m3t1p3β1	19.51	19.24	19.46
138	m3t1p3β2	19.22	19.67	19.81
139	m3t1p3β3	21.45	20.04	20.52
140	m3t1p3β4	21.97	20.55	20.78
141	m3t1p4β1	17.63	17.82	17.07
142	m3t1p4β2	18.41	18.22	18.15

143	m3t1p4β3	19.02	18.51	18.73
144	m3t1p4β4	19.89	19.18	19.76
145	m3t2p1β1	15.63	15.32	15.41
146	m3t2p1β2	16.13	16.37	16.74
147	m3t2p1β3	17.27	16.85	17.13
148	m3t2p1β4	17.32	17.01	17.24
149	m3t2p2β1	19.16	19.21	19.07
150	m3t2p2β2	21.22	20.54	20.83
151	m3t2p2β3	22.17	21.26	21.12
152	m3t2p2β4	23.25	22.51	22.18
153	m3t2p3β1	24.84	24.11	25.17
154	m3t2p3β2	26.92	25.84	25.44
155	m3t2p3β3	28.55	27.92	27.19
156	m3t2p3β4	29.11	28.97	29.03
157	m3t2p4β1	21.43	20.87	21.91
158	m3t2p4β2	22.72	21.43	22.31
159	m3t2p4β3	23.97	23.19	23.46
160	m3t2p4β4	24.99	23.82	23.64
161	m3t3p1β1	17.54	17.88	17.32
162	m3t3p1β2	18.22	18.84	18.43
163	m3t3p1β3	19.76	19.36	20.08
164	m3t3p1β4	21.11	20.87	21.16
165	m3t3p2β1	23.71	23.44	22.95
166	m3t3p2β2	25.23	25.09	25.72
167	m3t3p2β3	26.19	26.76	26.50
168	m3t3p2β4	27.91	27.84	28.67
169	m3t3p3β1	30.04	30.25	30.61
170	m3t3p3β2	31.87	31.23	31.73
171	m3t3p3β3	32.18	32.64	32.81
172	m3t3p3β4	34.27	33.92	34.19
173	m3t3p4β1	22.74	21.71	22.15
174	m3t3p4β2	23.12	22.17	22.34
175	m3t3p4β3	24.21	24.04	23.61
176	m3t3p4β4	26.91	25.11	25.03
177	m3t4p1β1	16.74	16.23	16.89
178	m3t4p1β2	17.49	16.54	17.91
179	m3t4p1β3	17.23	18.02	17.68
180	m3t4p1β4	17.72	17.63	17.87
181	m3t4p2β1	19.75	19.22	19.03
182	m3t4p2β2	20.78	19.93	20.14
183	m3t4p2β3	21.09	20.73	20.91

184	m3t4p2β4	21.85	21.23	20.98
185	m3t4p3β1	23.23	22.72	23.46
186	m3t4p3β2	21.26	21.07	22.19
187	m3t4p3β3	21.41	22.72	22.29
188	m3t4p3β4	22.22	22.58	23.04
189	m3t4p4β1	23.10	23.21	23.53
190	m3t4p4β2	22.21	21.73	21.92
191	m3t4p4β3	22.02	21.61	21.43
192	m3t4p4β4	21.87	21.35	21.26
193	m4t1p1β1	9.85	10.12	9.64
194	m4t1p1β2	9.97	10.23	9.71
195	m4t1p1β3	9.72	10.76	10.53
196	m4t1p1β4	9.88	10.34	10.21
197	m4t1p2β1	11.24	11.71	12.07
198	m4t1p2β2	11.71	12.80	12.62
199	m4t1p2β3	11.98	12.93	12.44
200	m4t1p2β4	12.54	13.08	13.22
201	m4t1p3β1	14.27	15.16	14.83
202	m4t1p3β2	14.75	15.22	15.60
203	m4t1p3β3	14.99	15.77	17.03
204	m4t1p3β4	16.22	16.31	17.07
205	m4t1p4β1	13.01	13.87	13.46
206	m4t1p4β2	13.33	13.65	14.12
207	m4t1p4β3	14.12	14.08	14.81
208	m4t1p4β4	14.87	15.24	15.19
209	m4t2p1β1	10.11	10.94	10.72
210	m4t2p1β2	11.27	12.01	11.97
211	m4t2p1β3	11.82	11.91	12.74
212	m4t2p1β4	12.01	12.18	12.50
213	m4t2p2β1	13.67	13.22	13.81
214	m4t2p2β2	15.32	15.19	14.28
215	m4t2p2β3	17.12	16.91	16.56
216	m4t2p2β4	17.94	17.22	17.67
217	m4t2p3β1	16.17	16.67	16.01
218	m4t2p3β2	18.79	17.34	18.73
219	m4t2p3β3	20.22	19.11	20.76
220	m4t2p3β4	21.17	21.83	22.71
221	m4t2p4β1	15.74	15.22	15.51
222	m4t2p4β2	17.61	16.91	16.34
223	m4t2p4β3	19.37	18.17	18.76

Where: m=moisture content, t=temperature, p=pressure, t= heating duration

3.4. Analysis of Variance of oil yield

The results for the analysis of variance of oil yield is presented in Table 4.

Table 4: Analysis of Variance for Oil Yield

Source	Sum of Squares	Df	Mean Square	F	Sig.	Partial Eta Squared
M	8785.57	3.00	2928.53	15700.00	0.000	0.989
ϴ	6484.75	3.00	2161.58	11590.00	0.000	0.985
P	7935.16	3.00	2645.05	14180.00	0.000	0.988
t	638.89	3.00	212.96	1141.00	0.000	0.870
mϴ	327.47	9.00	36.39	195.02	0.000	0.774
Mp	172.45	9.00	19.16	102.70	0.000	0.644
ϴp	355.41	9.00	39.49	211.65	0.000	0.788
ϴt	216.41	9.00	24.05	128.88	0.000	0.694
mϴp	273.38	27.00	10.13	54.27	0.000	0.741
Error	95.53	512.00	0.19			
Total	25285.01	587.00				

a. R Squared = .996 (Adjusted R Squared = .994)

b. F_{tab}= 3.84 5% level of Significance

*m=moisture content, ϴ=temperature, p=pressure, t= heating duration

From Table 4, it can be inferred that the process conditions (moisture content, heating temperature, pressure and heating duration) and their interactions had significant effect on oil yield at 5% level of significance. This implies that at least one of the mean treatment effects is significantly different from the others. The Partial Eta Square (PES) statistics shows the 'practical'significance of each process condition. Larger values of PES indicate a greater amount of variation accounted for by the model process conditions to a maximum of 1, i.e. the closer value of PES of process condition is to 1, the higher the *contribution* or *effect* of such term to the significance of the model. Hence it can be concluded from Table 4 that all the process conditions and their interactions contributed strongly to the significance of the model.

3.5 Effect of moisture content on oil yield

Duncan's New Multiple Range Test (DNMRT) was conducted to determine the differences in the mean treatment effect of moisture content on oil yield as reflected in the ANOVA table (Table 4) and this is shown in Table 5.

Table 5: Duncan's New Multiple Range Test for Oil Yield at various Moisture Contents

Moisture content (% wb)	Oil yield (%)
16.6	15.62a
13.2	21.21b
6.3	22.37c
8.1	24.86d

Means with the same alphabet are not significantly different from each other

Table 5 showed that seeds with higher moisture content yielded less oil as compared to seeds with lower moisture content. This agrees with the findings by Farsie and Singh (1985) who reported maximum oil recovery for sunflower seeds expressed at 6% moisture content. The authors reported further that increasing the moisture content to 14% decreased the oil recovery by 16%. However, from Table 5, it is observed that moisture contents at each level recorded significantly different oil yields. The maximum oil yield of 24.86% was observed at 8.1% mc while the minimum oil yield of 15.62% was recorded at the highest moisture content of 16.6%. Considering only the single effect of moisture content on oil yield. Both two-way and three way interactions of the process conditions were also significant indicating that the process conditions are highly interactive in nature.

3.6 Effect of heating temperature on oil yield

It can be inferred from Table 4 (ANOVA on oil yield) that heating temperature and its interactions with moisture content, pressure and heating duration had significant effect on oil yield at 5% level of significance. It implies that they all contributed significantly to the oil yield obtained.

The result of the Duncan's New Multiple Range Test (DNMRT) which was conducted to determine the differences in the mean treatment effect of heating temperature on oil yield is shown in Table 6.

Table 6: Duncan's New Multiple Range Test for Oil Yield at various Temperatures

Temperature	Oil yield
55	17.24a
100	20.06b
70	21.44c
85	25.31d

Means with the same alphabet are not significantly different from each other

Table 6 (oil yield at various temperatures) which shows the comparison among the four levels of heating temperature revealed that at any particular heating temperature, the observed means of oil yield are significantly different from each other. Table 6 further revealed that high oil yield was obtained at 85°C while the lower oil yield was obtained at 55°C. Considering only the single effect of temperature. This trend compares favourably with previous researches on oil expression by Adeeko and Ajibola (1990), Fasina and Ajibola (1989) Ajibola *et al*. (1990b), Fasina and Ajibola (1990), and Tunde-Akintunde*et al*. (2001). According to Adeeko and Ajibola (1990); Fasina and Ajibola (1989), heat treatment of oilseeds prior to oil extraction ruptures oil cells, reduces oil viscosity and increases flowability. These processes increase oil yield and extraction efficiency by allowing oil to flow easily during extraction (Olaniyan, 2010b); thus, this explains the increase in oil yield as the heating temperature was increased from 55 to 85°C in this study. Ayenew (2000) reported that oil recovery from Niger seed samples were influenced by increasing heat levels, and that the optimum oil recovery was obtained when the Niger seed samples were heated at 90^0C prior to expression.

Soetaredjo *et al*. (2008) however reported that preheating of neem seeds prior to oil expression resulted in lower yield of oil. They showed that as the preheating temperature increased, the yield of neem oil decreased, although they reported that this decrease was mainly related to the moisture content and structure of the seeds. Adejumo *et al.* (2013) reported the highest oil yield of 33.7% for moringa oleifera seed oil when the extraction was done at 100°C as against the 32.2 and 30.90% obtained at 130 and 150°C respectively.

3.7. Effect of heating duration on oil yield

From the result of the Analysis of variance for oil yield presented in Table 4, it can be inferred that heating duration and its interaction with heating temperature had significant effect on oil yield at 5% level of significance. The result of the Duncan's New Multiple Range Test (DNMRT) which was conducted to determine the differences in the mean treatment effect of heating duration on oil yield is shown in Table 7.

Table 7: Duncan's New Multiple Range Test for Oil Yield at various Heating Durations

Heating duration	Oil yield
5	19.77a
10	20.64b
15	21.42c
20	22.23d

Means with different alphabets are significantly different from each other

Table 7 (oil yield at various heating durations) which shows the comparison among the four levels of heating duration revealed that mean oil yields at the different levels were significantly different from each other. Longer heating duration yielded more oil as compared to shorter heating duration. This suggests that for more oil yield, the heating duration should be increased as much as possible, provided that other important qualities of the oil are not compromised. The highest heating duration (20 min) for this experiment recorded the highest oil yield of 22.22% which is statistically higher than all other heating durations used in this experiment. Considering only the single effect of heating duration

Olaniyan (2011) reported that maximum oil yield was extracted from orange seeds at 20 min roasting duration which was the highest duration used in the extraction process. Tunde-Akintunde *et al.* (2001) reported that oil yield of mechanically expressed soybean oil increased with increase in heating time from 15 to 30 min; the authors observed that the highest oil yield of 10.4% was obtained when the samples were heated for 30 min at a temperature of 80°C. Manpouya *et al.* (2013) and Jing *et al.* (2012) observed that extraction time of 2 and 3 h gave optimum oil yield in the solvent extraction of safou pulp (*Dacryodes Deulis*) and rape seed oils. Awolu *et al.* (2013) reported that increasing the extraction time and particle size of neem seed mass from 1 h and 0.42 mm to 2 h and 1.39 mm, respectively, gave maximum oil yield in the solvent extraction of oil from neem seed.

Adeeko and Ajibola (1990) reported that the rate of oil expression from groundnut seeds increased with increase in heating duration and heating temperature when moisture content and pressing pressure are held constant. Bamgboye and Adejumo (2011) reported an increase in rosselle oil yield as the heating time was increased from 15 min to 25 min.

3.8 Effect of machine pressure on oil yield

From the ANOVA table for oil yield presented in Table 4, it can be inferred that applied pressure and its interactions had significant effect on oil yield at 5% level of significance. The partial eta squared (PES) statistics reported the ‘practical’ significance of this process condition. Hence, it was concluded based on data from Table 4 that applied pressure and its interactions contributed strongly to the significance of the process. Duncan’s New Multiple Range Test (DNMRT) was therefore conducted to determine the differences in the mean treatment effects of applied pressure on oil yield (Table 8).

Table 8: Duncan’s New Multiple Range Test for Oil Yield at various Pressures

Pressure (KN/m^2)	Oil yield (%)
602.91	16.18a
723.07	20.52b
1334.88	22.38c
925.84	24.99d

Means with different alphabets are significantly different from each other

The result of the comparison among the four levels of applied pressure revealed that each level of pressure recorded significantly different oil yield. Higher pressure seems to yield higher oil and becoming a maximum at 925.84 KN/m^2. Further increase in applied pressure beyond this point (925.84 KN/m^2) led to a reduction in yield. The highest oil yield of 24.99% was recorded at 925.84 KN/m^2 while the minimum oil yield of 16.18% was observed at 602.91 KN/m^2.

Soetaredjo *et al*. (2008) reported that seed particles will deform and compactly fill up empty voids at low pressure level, but when the pressure is increased, the voids will diminish and the seed particles begin to resist the applied pressure through contact points between particles. Further increase in pressure will force the oil to start flowing out of the particles and the authors concluded that the neem oil point appeared to be at 13789.51 KN/m^2, and that 34473.79 KN/m^2 is the optimum pressure since further pressure beyond this (such as 41638.54 KN/m^2) gave insignificant increase on the oil yield. Adeeko and Ajibola (1990) reported that this was so because empty voids between particles from which the oil could flow out were becoming smaller at higher pressures.

Olaniyan and Oje (2007) reported that oil yield of shea butter from shea kernel increased progressively with increase in applied pressure from 1.5 to 8.8 MPa. Ebewele *et al*. (2010) also reported a consistent increase in oil yield of rubber seed oil when the pressure was increased from 5 MPa to 8 MPa.

The trend observed in this study also agrees with the work of the following researchers: Adeeko and Ajibola (1990), who reported that Oil yield from groundnut seeds increased with increase in pressure up to 20 MPa beyond which the yield levelled off; Ajibola *et al*. (1990a) reported a significant increase in oil yield from melon seeds when applied pressure was increased from 5 to 18 MPa but oil yield either levelled off or decreased slightly when the pressure was increased to 25 MPa; Fasina and Ajibola (1989) reported an increase in oil yield from conophor nuts as pressure increased from 10 MPa to 25 MPa. This behaviour can be explained as follows: During the process of oil expression from oilseeds, increasing the pressure applied during screw pressing tends to decrease the size of the capillaries through which oil flows and further increase in pressure may eventually lead to the sealing of the capillaries and some inter kernel voids (Ward, 1976; Adeeko and Ajibola, 1990).

3.9 Model Developed

The coefficients of determination, R^2 for the response, yield, was 0.907. The model relating the process condition and output is given below:

Oil Yield

$$OY = -1.130 \times 10^{2} + 3.741\text{m} + 1.775\Theta + 1.005 \times 10^{-1}p + 1.631 \times 10^{-1}\text{t} - 1.644m^2 - 5.932 \times 10^{-3}m\theta + 2.661 \times 10^{-4}mp + 1.050 \times 10^{-2}\theta^2 - 4.595 \times 10^{-5}p^2 R^2_{adj} = 90.4\% \qquad 5$$

*Ө= Temperature, m= moisture content, t= heating duration, p= pressure

From the model equation generated, the following can be deduced:

Temperature (Ө)

Temperature had positive linear effects on oil yield. It also had positive quadratic effects on oil yield. The interactions between temperature and moisture content, temperature and pressure, temperature and heating duration had positive effect on oil yield.

Heating Duration (t)

Heating duration had positive linear effect on oil yield

Moisture Content (m)

Moisture content had positive linear effect on oil yield; it had negative quadratic effect on oil yield. The interactions between moisture content and pressure and moisture content and temperature had negative effects on oil yield.

Pressure (p)

Pressure had positive linear effect on oil yield; it had negative quadratic effect on oil yield.
Regression statistics was used to check the model adequacy. The adjusted coefficient of determination which defined the percentage of total variability explained by the models was 90.4%, oil yield. This high percentage of total variability explained by the model implies good fit. The values of coefficient of determination (R^2, 90.7%) and adjusted coefficient of determination (90.4%) are relatively close for all process parameters. According to David *et al.* (1998), this is what should be expected of good models.

4. Conclusions

The data generated from this study have been used to show that temperature, heating duration, moisture content, pressure and their interactions all proved to determine the quantity of oil yield from neem seed kernels. The mathematical model developed is adequate to relate the process input to the output. The reliability and adequacy of the model developed to do this was examined using the coefficient of determination R^2 and adjusted coefficient of determination (R^2). The results obtained indicated that the model was statistically adequate and can be used to relate process input to process output. The coefficient of determination and adjusted coefficient of determination (R^2) values were quite high (90.7 and 90.4%), this high percentage of total variability indicate goodness of fit.

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