



Effect of Roasting Temperature and Duration on the Quality of Oil from Walnut (*Plukenetia conophorum*) Using Response Surface Methodology

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Authors' contributions

This work was carried out author RFA with supervision of author AR. Author RFA performed the statistical analysis, wrote the protocol, wrote the first draft, managed the analyses of the study and managed the literature searches. Author AR designed the study and supervised the work. Both authors read and approved the final manuscript.

Article Information

DOI: 10.9734/AFSJ/2019/v13i430116

Editor(s):

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- Complete Peer review History: <http://www.sdiarticle4.com/review-history/53302>

Original Research Article

Received 01 November 2019
Accepted 05 January 2020
Published 13 January 2020

ABSTRACT

This study investigated the effect of heat treatment and duration on yield and qualities of Walnut (*Plukenetia conophorum*) oil. The experimental design was carried out using the central composite rotatable design (CCRD) of response surface methodology with a total of thirteen experimental runs. The process variables and their ranges were roasting temperature 87.57°C to 172.43°C and roasting duration 10 min to 46.21 min. Oil from each experimental run was extracted using Piteba screw oil expeller. The expressed oil yield was determined and the results revealed that the oil yield ranged from 33.87% to 30.67% for the maximum and minimum values which were achieved at temperatures and duration of 172.43% at 25 min and 87.57°C at 25 min respectively. Optimum process condition was achieved at roasting temperature and duration of 160°C and 40 min with two possible solutions of 0.82 desirability which gave oil yield (33.50%); peroxide value (7.8 MeqO₂/g);

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colour (13.69 mg/L); free fatty acid (1.46%) and iodine value (136.82 mmol/100 g). The low free fatty acid and peroxide values in walnut oil is an indication of its overall quality thus its suitability as an alternative oil to supplement existing edible oils.

Keywords: Walnut oil; heat treatment; oil yield; free fatty acid; iodine value and peroxide value.

1. INTRODUCTION

Oil is an important component of food plant. It is found along with other components such as protein and a wide variety of carbohydrate (cellulose, hemicelluloses, starch) in plant cell [1]. The oil content of vegetable oil-bearing materials varies between 3 and 70% of the total weight of the seed, nut, kernel or fruit. Oil content of various seeds vary and this serves as a basis for the classification of seeds into oilseeds and non-oilseeds. Seeds with content greater than 17% are considered as oilseeds [2]. Oilseed crops are grown primarily for the oil they contain. They are very important component of modern agriculture for they provide easily available and highly nutritious human and animal food [3]. Nutritionally oils obtained from oilseeds provide the calories, vitamins and essential fatty acid in the human diet in an easily digestible form and at a relatively low cost [4]. In Nigeria, among the Yoruba tribe, Walnut is known as *asala* or *awusa*, *ukpa* or *okeokpokirinya* in igbo; and *gawu dibairi* in Hausa and it is called *okhue* or *okwe* among the Bini tribe of Edo state [5,6]. It is oil yielding crop plant which is very important for economic growth in the agricultural sector. Ripe Walnuts are mostly consumed fresh, toasted or used in cakes, desserts and confectionaries. These seeds are edible even when raw. Walnuts are rich in fat. A diet supplemented with Walnuts has a beneficial effect on blood lipids and lowering of blood cholesterol [7].

It also contains considerably high levels of non-cholesterol sterols known as plant sterols or Phytosterols [8]. Black walnuts contain higher levels of phytosterols (109 mg/100 g) [9]. Phytosterols are able to reduce cholesterol absorption in the intestinal lumen due to their high affinity for forming micelles. Consequently, cholesterol is displaced from micelles resulting in lower cholesterol absorption and reduced plasma cholesterol [10]. The supplementation of a background diet with 68 g of walnut/day has been discovered to reduce the total and low-density lipoprotein cholesterol by 5 and 9% respectively, and it was suggested that these reductions would have some positive effects in reducing the risk of coronary heart disease [11]. Torabian et al. [12] discovered that

supplementing walnuts for ~12% of total daily energy intake without changing habitual diet decreased total cholesterol and triglyceride levels. The rate of vegetable oil consumption is increasing compared with animal fat due to its health implication [3]. The industry is thus challenged to produce high quality oil products at reduced price. Importance of oil crops as a vital part of the world's food supply is evidenced in world agricultural trade statistics [13]. In Nigeria, some of the major oils usually consumed are palm oil, groundnut oil and coconut oil. These conventional sources of vegetable oil have been of great significance over the years for both human and industrial use with increase demand because of the growing rate of population. Therefore, the need exists to look for other sources to supplement the supplies especially underutilized oilseeds [14]. A number of studies have been done on the walnut seed such as the determination of oxalate, phytates and tannins [15]. Edem, et al. [16] also reported on the proximate composition, ascorbic acid and heavy metal contents of the nut. The aim of this work was to study the effect of roasting temperature and duration on the yield of Walnut oil and characterize some quality parameters of the oil produced.

2. MATERIALS AND METHODS

2.1 Materials

African Walnuts (*Pluckennetia conophora*), were purchased from Jakande fruits and vegetable market in Ketu, Lagos, Nigeria. All the chemicals used in this work were of analytical grade from BDH Chemical Laboratory (England, UK).

2.2 Methods

2.2.1 Sample preparation

The nuts were washed to remove dirt and possible external contaminants. They were sundried for 48 h to facilitate easy cracking of the shell which was done manually. These nuts were thereafter reduced to smaller sizes using a mixer grinder (550 W Speedo) prior to roasting in an oven (N53CF model) at a specified temperature-duration combination. The method reported by Akinoso, et al. [17] with slight modification was

used. The samples initial temperatures were raised to equilibrium with roasting temperature. This was achieved by wrapping them in foil paper and placed in the oven at desired roasting temperature level and duration combination as outlined in Table 1. For each treatment combination, about 100 g of ground sample was thinly spread in a petri-dish and placed in a pre-set oven at different temperatures and duration combinations prior to extraction of the oil.

2.2.2 Experimental design

A central composite rotatable design of Response Surface Methodology (RSM) was used as described by Montgomery [18]. The independent variables of roasting temperature and roasting duration whose ranges are from 100°C to 160°C and 10 minutes to 40 minutes respectively were used to generate a design matrix as shown in table consisting of thirteen experimental combinations. The responses for this design are oil yield, free fatty acids, iodine value and peroxide value. The experimental analyses were carried out in triplicate and the mean values were recorded as obtained data. Data were analyzed using Design Expert – Version 6.0.6 (Stat Ease Minneapolis, USA) software package to generate regression equations. Analysis of variance (ANOVA) was determined at 5% level of significance. Coefficients of determination (R^2) values and lack of fit test were used to determine the suitability of the models.

2.2.3 Oil extraction

A modified method by Popoola, et al. [19] was used. The walnut oil was expressed using handmade Piteba screw oil expeller (Holland). It is a manually operated expeller, with the barrel heated for about 10 min before loading of the pre-heated samples into the expeller. The screw moved the sample toward the press cage outlet when the handle was turned. As a result of accumulation of sample toward the press cage outlet, the sample was further ground and exposed to a very high pressure. With the help of the continuous heat supplied, the oil was expelled near the press cage outlet and ran against the direction of flow of the sample.

2.2.4 Analysis

2.2.4.1 Determination of oil yield

The oil yield was calculated from the weight of extracted oil to the weight of the sample according to Equation 1.

$$\% \text{ Oil Yield} = \frac{\text{Weight of extracted oil}}{\text{Weight of sample}} \times 100 \quad (1)$$

2.2.4.2 Determination of peroxide value

The peroxide value was determined according to a method described by AOAC, [20]. In this method, Oil sample (0.5 g) was added into a boiling tube containing 1 g powered potassium iodide. Glacial acetic acid/chloroform mixture (20 mL; 2:1) was added, the boiling tube was placed in boiling water for 1 min after which its content was poured into conical flask containing potassium iodide solution (20 mL; 5%). The boiling tube was rinsed twice with distilled water (25 mL) and content added into the conical flask. The whole content was titrated with sodium thiosulphate (0.002 M) solution to colourless end point using starch as indicator. Results are expressed as mmol/kg. Peroxide value of the oil sample was calculated accordingly (Equation 2).

$$\text{Peroxide value} = \frac{(V_s - V_b) \times \text{molarity of titrant}}{w} \times 100 \text{ g Kg}^{-1} \quad (2)$$

Where

V_b = Titre for blank;

V_s = Titre for sample;

W = Weight of sample in grams.

2.2.4.3 Determination of iodine value

Determination of iodine value (Wijs' method) was according to AOAC [20]. The sample (2%) was prepared in chloroform, titrated with Wijs' solution (5 mL) mixed thoroughly and allowed to stand in the dark for 5 min. Potassium iodide solution (5 mL; 7.5%) was added and titrated to a light straw colour using 0.1 N sodium thiosulphate solution. Starch indicator (3 drops) was thereafter added and titration continued to a colourless (white or milky) end point. Results are expressed as I₂ 100/g. Iodine value of the oil sample was calculated (Equation 3)

$$\text{Iodine value} = \frac{(V_b - V_s) \times 1.269}{w} \quad (3)$$

Where

V_b = Titre for blank;

V_s = Titre for sample;

W = Weight of sample in grams.

2.2.4.4 Free fatty acid determination

Free fatty acid was calculated using AOCS method Ca5a-40 [21]. A mixture of 1.0 g of oil

was dissolved into 25 mL of diethyl ether and 25 mL of alcohol after which 1 mL of phenolphthalein solution was used as an indicator. The mixture was titrated with aqueous 0.5 N NaOH. There was a continuous shake until a permanent faint pink colour was observed which lasted for more than 15 s. The % free fatty acid was then calculated as

$$\% \text{ FFA} = \frac{\text{Titration (mL)} \times 0.14}{\text{Weight of sample}} \times 100 \quad (4)$$

Table 1. Central composite rotatable experimental design

Experimental runs	Roasting temperature (°C)	Roasting duration (min)
1	160.00	40.00
2	130.00	25.00
3	130.00	25.00
4	100.00	40.00
5	130.00	46.21
6	100.00	10.00
7	87.57	25.00
8	160.00	10.00
9	130.00	25.00
10	130.00	25.00
11	172.43	25.00
12	130.00	25.00
13	130.00	3.79

2.2.4.5 Determination of colour

The color of the oil was determined according to AOCS Cc 13c-50 standard methods [22] with the aid of a North Star Scientific spectrophotometer (UVIKON XL, UK).

3. RESULTS

3.1 Oil Yield

Significant difference ($p < 0.05$) of the effect of heat treatment on oil yield was observed in this study. The range of oil extracted from the ground walnut (1000 g) was equivalent to 33.87% and 30.67% which was achieved at temperature and duration of 172.43°C at 25 min and 87.57°C at 25 min respectively (Table 2). The 3D response surface plots of the interaction are shown in Fig. 2.

3.2 Peroxide Value

The effect of both roasting temperature and duration was significant ($p < 0.05$) on the peroxide

value of the oil with the minimum and maximum value being 7.5 MeqO₂/g and 9.7 MeqO₂/g respectively (Table 2). The coefficient of determination (R^2) of 0.9764 showed that the model fits well for the data (Table 3); and 3D response surface plots of the interaction as shown in Fig. 2 as well as the quadratic model best describe the relationship between the roasting temperature and duration and the oil yield (Table 4).

3.3 Iodine Value

The effect of both roasting temperature and duration was significant ($p < 0.05$) on the iodine value of the walnut oil. The minimum value was 129.91 mmol/100 g while the maximum value 168.14 mmol/100 g (Table 2). This result is in agreement with the contour and response surface plot of the interaction presented in Fig. 3. There was significant difference ($P < 0.05$) in the effect of both roasting temperature and duration on iodine value of the walnut oil. The coefficient of determination (R^2) of 0.9577 showed that the model fits well for the data (Table 3). Also, 3D response surface plots of the interaction as shown in Fig. 3 and the quadratic model best describe the relationship between the roasting temperature and duration of the oil yield (Table 4).

3.4 Colour

Colour intensity of the oils ranged from 5.59 mg/L at 87.57°C for 25 min to 13.82 mg/L at 172.43°C for 25 min at roasting temperature and duration of the oil. The oil color was significantly ($p < 0.05$) dependent on heat treatments. The 3D response surface plot is represented in Fig. 5.

3.5 Free Fatty Acid

The effect of both roasting temperature and duration was significant ($p < 0.05$) on the free fatty acid of the walnut oil with the minimum and maximum value of the oil recorded at 1.25% at 172.43°C for 25 min and 2.11% at 87.57°C for 25 min roasting temperature and roasting duration respectively. Fig. 4 represents the effect of roasting temperature and duration on the free fatty acid value of the walnut oil presented in the 3D response surface plots interaction.

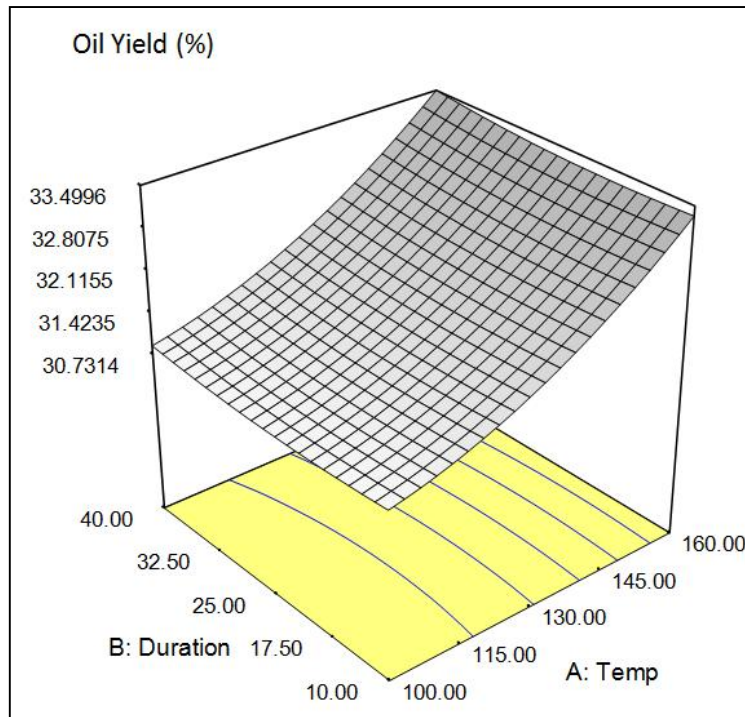


Fig. 1. 3D response surface plots of the effect of roasting temperature and roasting duration on the oil yield (OY) of walnut oil

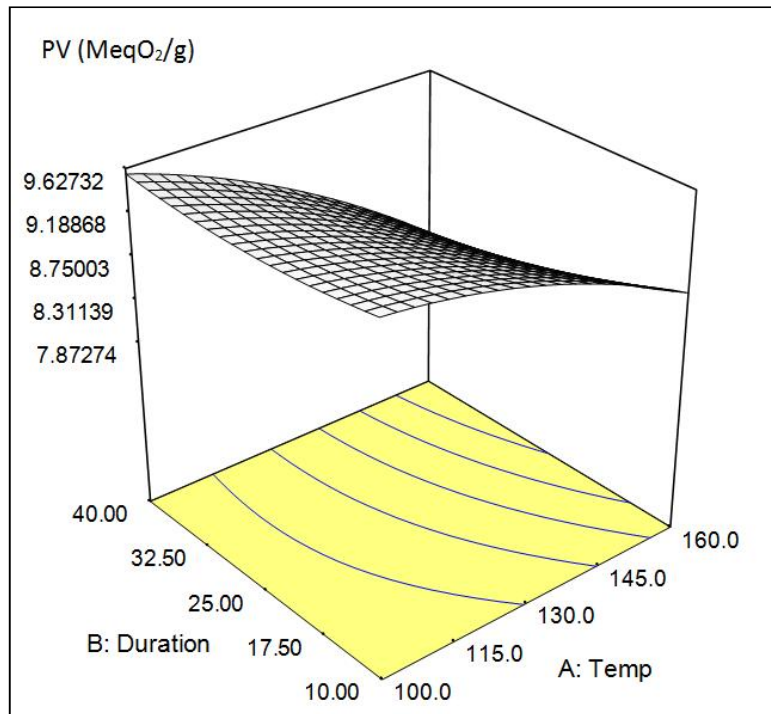
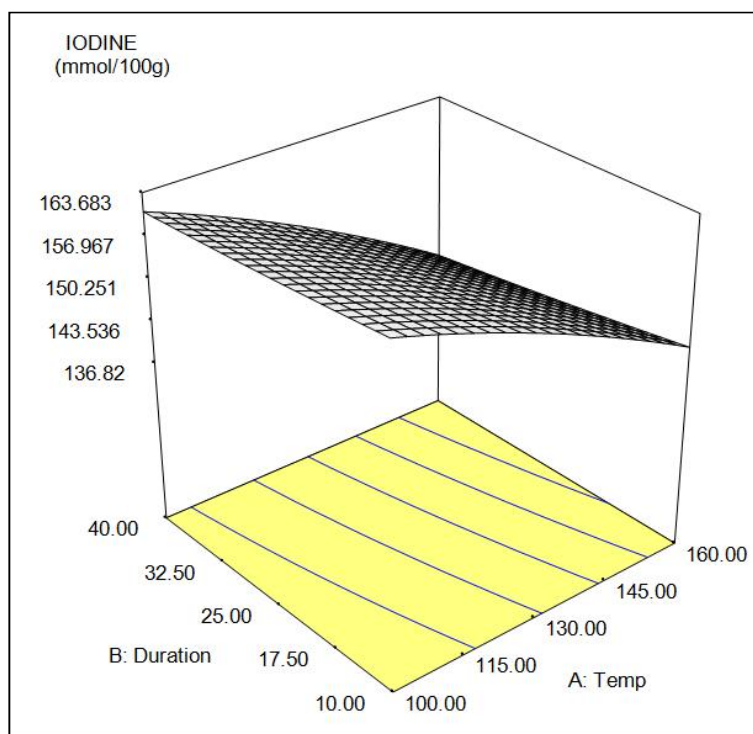


Fig. 2. 3D response surface plots of the effect of roasting temperature and roasting duration on the peroxide value (PV) of walnut oil

Table 2. Central composite rotatable design matrix of the experimental runs and their response values

Exp. Runs	Roasting temperature (°C)	Roasting duration (Mins)	Oil yield (%)	Peroxide value (MeqO ₂ /g)	Colour mg/L	Iodine Value (mmol/100g)	Free Fatty Acid(%)
1	160.00	40.00	33.77	8.0	13.48	139.59	1.51
2	130.00	25.00	31.64	9.0	11.80	152.28	1.77
3	130.00	25.00	31.64	9.0	11.80	152.18	1.77
4	100.00	40.00	30.82	9.5	10.18	158.83	1.93
5	130.00	46.21	31.83	8.9	12.65	149.11	1.74
6	100.00	10.00	30.82	9.6	8.27	161.80	2.00
7	87.57	25.00	30.67	9.7	5.59	168.14	2.11
8	160.00	10.00	33.69	8.8	12.74	145.94	1.59
9	130.00	25.00	31.64	9.0	11.80	152.28	1.77
10	130.00	25.00	31.64	9.0	11.80	152.28	1.77
11	172.43	25.00	33.87	7.5	13.82	129.91	1.25
12	130.00	25.00	31.64	9.0	11.80	152.28	1.77
13	130.00	3.79	31.56	9.4	11.70	155.45	1.80

**Fig. 3. 3D response surface plots of the effect of roasting temperature and roasting duration on the iodine value (IV) of walnut oil**

3.6 Models

Model equations to predict the effect of independent process variables (roasting temperature and roasting duration) on oil yield, peroxide value, iodine value, colour and free fatty acid of the walnut oil generated are presented in Table 3 from the empirical data obtained. The

coefficients of determinations (R^2) of regression models varied between 0.9577 and 0.9764. Based on lack of fit test and coefficient of determination (R^2), quadratic model was found suitable to all the responses i.e. oil yield (OY), peroxide value (PV), iodine value (IV), colour and free fatty acid (FFA) values of the walnut oil.

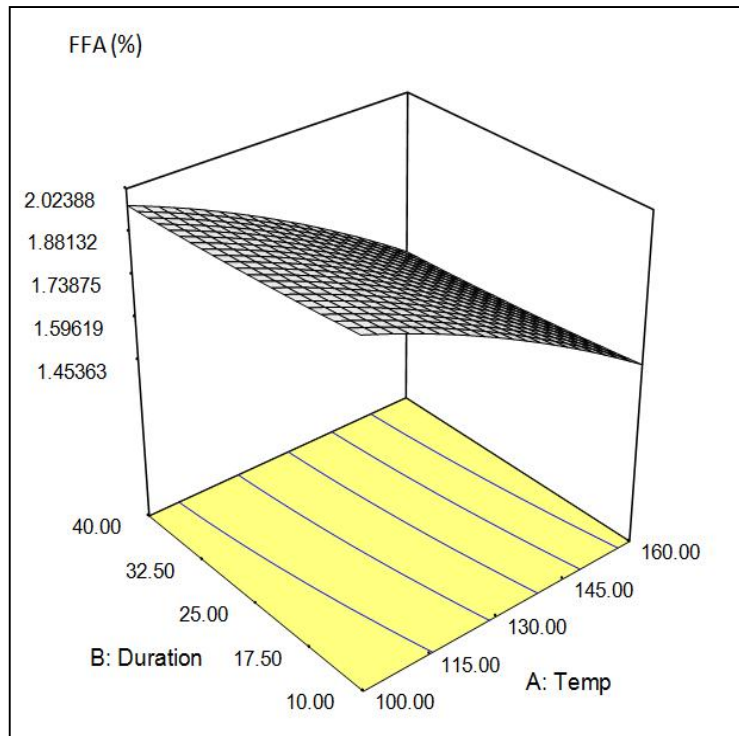


Fig. 4. 3D response surface plots of the effect of roasting temperature and roasting duration on the Free Fatty Acid (FFA) of walnut oil

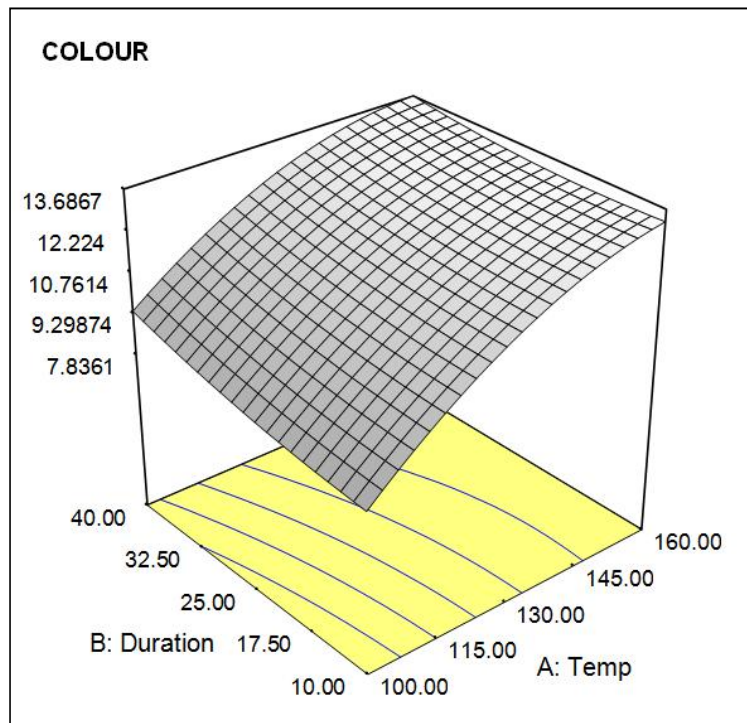


Fig. 5. 3D response surface plots of the effect of roasting temperature and roasting duration on the colour of walnut oil

Table 3. Analysis of variance (ANOVA) and the coefficient of determination (R^2) of the effect of roasting temperature and roasting duration on the walnut oil

Source of variation	Sum of squares of response variables					
	DF	Oil yield	Peroxide value	Iodine value	Colour	Free fatty acid
Model	5	14.47	4.43	1053.24	57.24	0.54
Roasting temp	1	13.38	3.66	993.83	49.09	0.52
Roasting duration	1	0.03	0.32	41.80	1.99	0.006895
A ²	1	1.05	0.21	13.55	6.83	0.009460
B ²	1	0.07	0.070	0.37	0.42	0.004594
AB	1	1.58898	0.12	2.86	0.34	0.002500
Residual	7	0.39	0.11	46.54	2.91	0.021
LOF	3	0.39	0.11	46.53	0.73	0.021
Pure Error	4	0.000	0.000	0.008000	0.000	0.000
Cor Total	12	14.86	4.53	1099.78	59.43	0.56
MFV		51.67	57.99	31.68	36.64	35.59
MPV		0.0001	<0.0001	0.0001	0.0001	0.0001
R ²		0.9577	0.9764	0.9577	0.9632	0.9622
Adjusted R ²		0.9275	0.9596	0.9275	0.9369	0.9351
PRESS		330.99	0.76	330.99	15.55	0.15

A = Temperature B = Duration LOF = Lack of fit MFV = Model for F value MPV = Model for P value
R² = coefficients of determination

Table 4. Predictive equation models for the effect of roasting temperature and duration of responses

S/N	Response	Regression model equations	Model type
1	Oil yield	OY = +31.64 +1.29xA+0.058xB+0.39 xA ² + 0.10xB ² + 0.020xAxB	Quadratic
2	Peroxide value	PV=+9.00-0.68xA-0.20xB-0.17xA ² +0.10xB ² -0.17xAxB	Quadratic
3	Colour	CO = +11.80+2.43xA+0.50xB-0.99	Quadratic
4	Iodine value	IV=+152.26-11.15x A-2.29x B-1.40x A ² +0.23xB ² -0.84xAxB	Quadratic
5	Free Fatty Acid	FFA = +1.77-0.26x A-0.029x B-0.037x A ² +8.128 E-003x B ² -0.002500x A x B	Quadratic

A = roasting temperature (°C) and B = roasting duration (min)

3.7 Optimum Parameters

Oil yield was maximized while peroxide value, iodine value and free fatty acid were minimized and the colour of the walnut oil was within range. The computer software package (Design Expert Version 6.0.6 Stat Ease Minneapolis, USA) for optimization for the best desirability was achieved at roasting temperature and duration of 160°C for 40 min with a predicted oil yield of 33.50%. The optimum oil yield (33.50%) is lower than the maximum oil yield (33.87%) obtained in this study.

4. DISCUSSION

The maximum oil yield value of 33.87% was at 172.43°C with 25 min roasting temperature and

duration respectively. The oil yield at 160°C for 40 min was increased (33.77%) while at the same temperature but at lower duration (10 min), the oil yield was decreased (33.69%). This also implies that at higher temperature oil yield is increased with time. This value (33.87%) was found to be lower than the oil yield reported by Wilfred, et al. [23] who worked on expression of oil and assessing the quality and stability of pressed and solvent extracted oil from *Allanblackia floribunda* seeds (48.60% from manual expeller). Akinoso [3] who worked on palm kernel also reported a maximum oil yield (47%) which is higher than that recorded in this work. However, the yield recorded in this study was found to be slightly higher than the maximum yield (32.6%) reported by Popoola, et al. [19] who worked on *egusi igba* bottle gourd

seed. This observed variation might be attributed to varietal differences in the oil plant as well as some other factors among which include variations in oil yield to plant variety, methods of oil expression, efficiency of the equipment, cultivation, climate, ripening stage and harvesting time of the seed among others, all of which allow easy flow of the oil [24,25,3]. This shows that variation of roasting temperature and duration affects oil yield. A response surface plot of the interaction is shown as Fig. 1. Generally, temperature and duration influenced the yield of oil because better extraction was achieved by heating at higher temperature and time due to the fact that it causes breakdown of oilseed cells, coagulation of protein, and releases oil from the intact cells which removes moisture and reduces the oil viscosity [26].

The peroxide value obtained is less than the FAO/WHO [27] standard for fresh edible oil of 10 meqO₂/g which implies that the oil is suitable for consumption. The significance of analysis of variance and regression at 95% confidence level was an indication that the response surface quadratic model appropriately expresses the relationship between roasting temperature and duration of walnut oil and the resultant peroxide value.

This study shows that peroxide value decreases with an increase in temperature as shown in Table 2. The lower peroxide value of *moringa* oil indicates its resistance to rancidity. The maximum peroxide value obtained here is slightly higher than that of *Moringa Oleifera* (8.10 meqO₂/g) but lower than the value obtained for palm oil (13.40 meqO₂/g) as reported by Ogbunugafor, et al. [28]. Peroxide value indicates the deterioration of oils; and oil with higher peroxide value indicates higher rancidity [29,30]. Generally, highly unsaturated oils are known to absorb more oxygen and develop higher peroxide values. WHO/FAO stipulated a permitted maximum peroxide level of not more than 10 M equivalent of peroxide oxygen/kg of the oils [30].

The formation of peroxide is a chemical reaction, and like most reactions, it is influenced by heat and some other factors [31]. Since peroxide value measures the initial stages of oxygen absorption in oil and is considered satisfactory at values ≤ 10 . Therefore, the oil in this study will be suitable for consumption since it has a peroxide value that is less than 10 meqO₂/g.

Iodine value is a measure of the degree of unsaturation in oil; and could be used to quantify the amount of double bonds present in the oil which reflects the susceptibility of oil to oxidation [32]. Iodine value is useful in predicting the drying property of oils (Orhevba, 2013). The values observed indicate that the oil obtained in this study can be regarded as semi drying and non-drying. Low iodine number implies the presence of few unsaturated bonds and hence low susceptibility to oxidative rancidity [33]. The values show that the iodine value of the oil decreased with increase in roasting temperature. Generally, the lower the iodine value, the lower the degree of unsaturation and hence the lower the tendency of the oil to undergo oxidative rancidity [34]. In this study, the free fatty acid increased with increase in temperature and time and only run 7 (2.11%) of all thirteen samples has FFA value slightly more than the permissible level of 2.00% as recommended by FAO/WHO [35] and Standard Organization of Nigeria [36]. This might be as a result of inactivation of lipase enzyme [4]. The maximum free fatty acid value obtained (2.11%) in this study was lower than the value 2.35% reported for camelina oil [37] and ginger bread plum (15.10%) [38]. The low level of free fatty acid of the samples suggests low level of hydrolytic and lipolytic activities [39,40]. Values obtained for walnut oil in this study suggests its suitability as edible oil as reduction in free fatty acid content of vegetable oil enhances the quality grade.

The minimum and maximum color of the oil was 5.59 to 13.82 (Table 2) at roasting temperature of 87.57°C and 172.43°C. The combined effect of the treatment was significant ($P < 0.05$) on color. The quadratic model was best used to predict the effect of the temperature and duration on the color of the oil samples (Table 4). The high coefficient of determination R² (0.96) is an indication that the model had a good fit. The color intensity of the oil increased with increase in roasting temperature and roasting duration (Table 2). Oil color is understood to be due to the presence of carotenoid and chlorophyll pigments [26]. This finding is similar to that reported by Akinoso and Raji [41] on effect of roasting conditions on the color of locust seed oil.

4.1 Models and Optimum Parameters

The coefficients of determination (R²) of regression model for oil yield, peroxide value, iodine value, colour and free fatty acid were 0.9577, 0.9764, 0.9577, 0.9632 and 0.9622

respectively (Table 4) indicating a good fitness. That implies that the models can be used to navigate the design space and can be used as predictive models with fewer errors at 5% level of significance. The regression analyses indicated that some of the quadratic terms were significant ($p < 0.05$) in many of the regression models derived.

The range for desirability is between 0 to 1, with it represents the closeness of a response to its ideal value. If a response falls within the unacceptable intervals the desirability is 0 and if it falls within the ideal intervals or the response reach as to ideal value, the desirability is 1. Two possible desirability solution was achieved with 0.82 suggested as the best. The optimum process condition was achieved at roasting temperature and duration of 160°C and 40 min which gave oil yield (33.50%); peroxide value (7.8 MeqO₂/g); colour (13.69mg/L); free fatty acid 1.46% and iodine value (136.82 mmol/100g). The oil yield (33.50%) is lower than the maximum oil yield (33.87%) obtained in this study. This is envisaged as a result of the adjustment made to achieve the conditions.

5. CONCLUSION

The following conclusions can be drawn from this study

- i. The yield of the Walnut oil increases with increase in roasting temperature and duration
- ii. The effect of heat treatment and duration were significant in all the responses evaluated.
- iii. The values of the oil yield and colour increases with increase in temperature while free fatty acid, peroxide value as well as iodine value decreases with increase in temperature.

Based on these results, walnut oil might be a potential use as plant oil in human diet.

6. RECOMMENDATION

The need for boiling of the walnut prior to extraction of the oil through heat treatment has become imperative due to the bitter taste usually encountered during walnut consumption which also reflected in the walnut oil. This bitter taste is as a result of some antinutrients which are present. Several studies have shown decrease in antinutritional factor in crops after heat treatment like blanching and boiling.

ACKNOWLEDGEMENT

The author acknowledges the contribution of Management of Federal Institute of Industrial Research, Oshodi, Nigeria for giving me the opportunity for further studies.

COMPETING INTERESTS

Authors have declared that no competing interests exist.

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