Assessment of the Quality of Bar Soaps Produced from Blends of Palm-Oil and African Walnut Seed-Oil

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ABSTRACT

This study was conducted to determine the characteristics of soap produced from various fats and oils. Palm oil was purchased, and walnut oil was extracted from its seeds. Five blends of palm oil and African walnut oil were used, together with their individual oils. The cold method of soap-making was employed in this study. Five blends (PW_0, PW_1, PW_2, PW_3, and WP_0) were prepared in a ratio, and each blend was used for soap production. Each oil blend was characterized for saponification value, acid value, peroxide value, iodine value, and free fatty acid content, and their results were as follows: (182±0.20 – 234±0.30 mgKOH/g), (1.60±0.20 – 3.37±0.12 mgKOH/g), (5.0±0.25 –12.5±0.1 Meq KOH/g), (37.5±0.50 – 55.6±0.25 gI_2100/g) and (0.80±0.20 – 1.68±0.12 mgKOH/g). The percentage of total fatty matter in the soap samples ranged from (52±0.1% to 77 ± 0.05%). WP_0 (palm oil 0% – walnut oil 100%) had the lowest percentage value of total fatty matter (52±0.1%) among the soap samples produced, while PW_0 (palm oil 100% – walnut oil 0%) had the highest percentage value of total fatty matter (77±0.05%). Judging by % total fatty matter, PW_0 gave the best soaps because its total fatty matter value fell within the acceptable limits of the Standard Organization of Nigeria (SON). The pH values observed from the blended samples ranged from 10.28 ± 0.01 – 12.20 ± 0.03. PW_3 (palm oil 25% – walnut 75%) had the highest values of 12.20 ± 0.03, which makes it harsh for the body. Thus, based on the findings of this study, it can be concluded that soap samples PW_0 (palm oil 100% – walnut 0%) and PW_1 (palm oil 75% – walnut 25%) can be suggested for use in laundry due to their favorable physicochemical properties (high% Total Fatty Matter, normal pH values, high foam ability).

Key Words: Soap, African Walnut, Palm Oil, Total Fatty Matter, pH

INTRODUCTION

Nuts have remained a major part of the diet for humans since pre-agricultural times. (King *et al.*, 2008). Hazel nut, macadamia, pecan, pistachio, cashew, almond, peanut, Brazil, and English walnut are some of the well-known and well-liked edible nuts (Bolling *et al.*, 2011). They can be consumed as meals or snacks. According to Rajaram and Sabate (2006), they are eaten whole (fresh or roasted), as spreads (peanut butter, almond paste), or as a component of commercial items like sauces, baked goods, oils, sauces, etc.

The African Walnut (*Tetracarpidium conophorum)* is a climbing shrub 10-20ft long. It is found mainly in Nigeria, Gabon, Equatorial Guinea Gambia, Sierra Leone, and Cameroon (Amaeze *et al.*, 2011). It is referred to as ukpa (Igbo) in Southern Nigeria and awusa or asala (Yoruba) in Western Nigeria. The seed is formed of two cotyledons wrapped in a hard shell-like casing, and the nuts are housed in pods that can have two to five nuts inside them. The nuts mature and are harvested between June and September, and are

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primarily processed in Nigeria by boiling in water or roasted in hot sand before being eaten as snacks (Nkwonta *et al.*, 2010). After consuming the nuts, drinking water frequently results in a bitter taste. This could be attributed to the presence of chemical substances such as alkaloids (Nkwonta *et al*., 2010).

The nut is widely used in decoctions in ethnobotanical medicine to treat a variety of illnesses, including dysentery, constipation, abdominal cramps, malaria, male sterility dysfunction, and general fever, as well as to manage chronic conditions such as diabetes, cancer, and high blood pressure. (Aladeokin and Umukoro, 2011; Amaeze *et al.*, 2011). The nut is extremely prone to fungal infestation; within a few days of harvest, as the pods decompose to release the nuts, they become moldy. The cotyledons become sticky, and fungal contamination is visible if the nutshells are cracked. (Amaeze *et al.*, 2011).

Palm oil is naturally reddish in color because of its high beta-carotene content. Red palm oil is named after its characteristic dark red color, which comes from carotenes, such as alpha-carotene, beta-carotene and lycopene, the same nutrients that give tomatoes, carrots, and other fruits and vegetables their rich colors. Red Palm Oil contains 10 other carotenes, tocopherols and tocotrienols (members of the vitamin E family), phytosterols, and glycolipids (Ping and Yuen, 2000).

A chemical molecule or mixture of chemical compounds known as soap can be described as the product of the reaction between fatty acids or fatty glycerides and a metal radical (or organic base). A soap may also be described as any water-soluble salt of fatty acids that contain eight or more carbon atoms. Sodium and potassium, which generate water-soluble soaps, are the most commonly used metals in soap production (Chalmers and Bathe, 1978).

Calcium soap has been utilized in animal feed composition, and soaps are often used for cleaning and laundry (Kuntom *et al*., 1994). The quantity and composition of the fatty acids in the starring oil define the characteristics of the soap. In each method used to make soap, blends of oils have always been used. By blending the separated fatty acids of palm oil (PO) and palm kernel oil (PKO), Kuntom (1996) generated soaps of desirable grade, and the quality of the soap produced was comparable to that of commercially available soaps. Therefore, this research aims to add to the scientific knowledge already established on the possible use of palm oil and African walnut oil in the manufacture of Bar Soaps.

MATERIALS AND METHODS

All chemicals and solvents used in the experimental work were of analytical grade (AR), and all commercial solvent samples were purified by the method reported in the literature (AOAC, 2005).

Samples collection and preparation

African walnuts “Asala or Awusa” (2kg) were purchased at terminus market in Jos, Nigeria. The kernel of the walnut was carefully separated from the seed. The immature seeds were then removed and later cut into small pieces (approximately 0.5 mm) to facilitate drying, and the sliced walnuts were first sun dried to reduce the moisture content of the fresh seeds. After sun-drying the nuts were sorted by removing decayed or defective seeds to ensure the quality of the extracted oil (Oloko, 2019).

Extraction of Walnut Seed Oil

For easier oil extraction, dried walnut seeds were ground into a powder using an electric blender. Oil was extracted from the samples using a solvent extraction method. 100g milled sample was wrapped with filter paper and then placed in a cellulose thimble in a soxhlet extractor, some n-hexane was placed in a round bottom flask up to two-third capacity full. The round-bottom flask was placed on a heating mantle, and temperature of the mantle was adjusted to 50-600C and evaporation occurred over a period of 8hrs. The extracted oil was reheated to remove n-hexane through evaporation (Oloko, 2019).

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Bleaching of Palm Oil

Palm oil (1.5kg) was placed in a strong saucepan material and allowed to boil for 2hrs using a charcoal stove. By inserting a piece of white paper into the oil, the paper turned oily and showed that the oil was fully bleached oil. The bleached Oil was allowed to cool and stored in a heat-safe plastic container (James, 2007)

Oil Blending

Oil blending was carried out in varying compositions as described by Eke *et al*., (2004). The blends were represented as shown below in Table 1

Table 1: Individual Oil and their Blend in Percentage Composition

Blend	Percentage Composition	
	P (%)	W(%)
PWo	100	0
PW_1	75	25
PW_2	50	50
PW_3	25	75
WP_0	0	100

P= Palm Oil, W= Walnut Oil

Determination of Physicochemical Properties of the Oil

Saponification Value

This represents the number of milligrams of KOH or NaOH required to saponify 1g of fat under specified conditions. The oil sample (2g) was weighed into a 500cm^3 conical flask and a 25ml pipette of 0.5M KOH solution was poured into the conical flask containing the oil. The reflux condenser in the flask was immersed in boiling water for one hour and two drops of phenolphthalein indicator were added after refluxing and titrated carefully with 0.5M hydrochloric acid. A blank (pottasium hydroxide solution without oil) was titrated using the same procedure. (Oloko, 2019; AOAC 2006)

$$\text{Saponification Value} = \frac{V * M * 56.1}{W} \quad (1)$$

A= Titrate of the sample

B= Titrate of the Blank

W= Weight of the sample, g

M= Molarity of HCl Solution

56.1= equivalent weight of potassium hydroxide

Acid Value

The Acid Value is defined as the number of milligrams of KOH needed to neutralize the organic acid present in 1g of fat and, is a measure of the free fatty acids present in the fat and oil. 25cm3 of diethyl ether with 25cm3 of ethanol was mixed and 1.0cm3 of 1% phenolphthalein solution neutralized the ethanol by

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titrating the solution with 0.1M KOH. 10g of oil sample was measured in a separate conical flask, neutralized ethanol was added, and the solution was boiled and heated until the sample neutralized the ethanol completely. The mixture was titrated against 0.1M potassium hydroxide and a few drops of phenolphthalein indicator was added (Obanla, *et al.,* 2018; AOAC, 2006).

$$\text{Acid Value} = \frac{V * M * 56.1}{W} \quad (2)$$

V= Volume of KOH required to neutralize the oil solution (ml)

M= Molar concentration of standard KOH

W= Weight of Oil sample (g)

56.1= Equivalent molecular weight of KOH

Acidity is frequently expressed as a free fatty acid for which the calculation is as follows:

Acid Value= Free fatty Acid x 1.99

Oil Yield

The oil was recovered by evaporation of the solvent on a heating mantle. The recovered oil was transferred to a beaker and placed in a water bath for complete evaporation of the solvent. The recovered oil was weighed (Obanla *et al*., 2018).

$$\% \text{ oil yield} = \frac{Z - Y * 100}{Z} \quad (3)$$

Z= Weight of the seed before extraction

Y= Weight of the seed oil after extraction

Iodine Value

The Iodine value is a measure of the degree of unsaturation of oil. It is the mass of iodine in grams consumed by 100g of chemical substance. The oil (0.5g) was weighed in a glass stoppard bottle. 10cm^3 of chloroform was added and dissolved, 20cm^3 of iodine solution was added and a stopper was inserted. The solution was allowed to stand in the dark for 30 minutes and 10cm^3 of 15% potassium iodide solution was added and shaken thoroughly, after which 100cm^3 of boiled and cooled water was used to wash down any free iodine in the stopper. The mixture was titrated against 0.1M sodium thiosulphate, a few drops of starch indicator were added, and a colourless white solution showed the end point of the titration. The same procedure was repeated for the blank titration (without oil samples) (Oloko, 2019; AOAC 2006).

$$\text{Iodine Value} = \frac{12.69\,(A - B)\,M}{W} \quad (4)$$

A = Volume in ml of standard sodium thiosulphate solution require for the blank

B = Volume in ml of standard sodium thiosulphate solution require for the sample

M = Molarity of standard sodium thiosulphate solution

W = Weight in gm of the sample

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Peroxide Value

The oil sample (1g) was weighed into a 250cm^3 Erlenmeyer flask with a glass stopper, 20cm^3 solvent mixture (acetic acid: chloroform, 3:2) and 1cm^3 of 1% saturated potassium iodide were added, followed by shaking for 1min, 30cm^3 of distilled water after shaking was added and shake again for 1min, the mixture was titrated against 0.025M $Na_2S_2O_3$ with vigorous agitation, 0.5cm^3 of 1% starch solution was added to the mixture with constant agitation to liberate iodine from the solvent layer. A blank titration was carried out without the oil (AOAC, 2006).

Peroxide Value = $\frac{S * M * 1000}{W}$ (5)

S= cm^3 of $Na_2S_2O_3$ (Test-Blank)

M= Molarity of standard sodium thiosulphate solution

W= Weight in gm of the sample.

GC-MS Analysis

SHIMADZU GC-2010 was used to examine the oil blends (PW_1, PW_2 and PW_3) using gas chromatography. The GC was outfitted with an auto-sampler (AOC-20s), an auto-injector (AOC-20i), and a capillary column with a film size of 30m x 0.25mm x 0.25m made by SH Rxi 5MS Sill. Helium was used as the carrier gas at a flow rate of 2.0 mL/min. The GC oven's initial temperature was 1400C for 10min, and then raised by 70C/min to a final temperature of 250^{0}C for 10min. The injection volume was 1.0 L with a 75:1 split ratio, and the injector temperature was 250^{0}C. The solvent had a 3.40-minute cutoff, and the entire run took 35.71 minutes. The SHIMADZU GCMS-QP-2020 detector was employed, at a temperature of 255^0 C (Olujuyigbe *et al*., 2019).

Preparation of Soap Samples (Saponification)

The cold process method was used for the saponification and the soap produced did not contain any additives (Muhammed and Usman 2018; Eke *et al*., 2004). The amount of oil used for saponification was 40 g, and each oil blend required a different amount of base (NaOH) to react completely, owing to the difference in the saponification value. The blended oil samples used for saponification were prepared by varying the weights of palm oil and walnut oil. For example, the blend containing 40g of palm oil and 0g walnut oil was labelled PW_0, and the blend containing 30g of palm and 10g of walnut oil was labelled PW_1. The blends yielded P: W ratios of 4:0, 3:1, 2:2, 1:3, and 0:4 (Table 1).

NaOH was dissolved in 16.5cm^3 of distilled water (alkali: water mixture is 25%), and 40g of the oil blend was slowly poured into NaOH solution. The solution was repeatedly stirred to form a liquid paste. The paste was stirred continuously until the paste thickness increased and a trace mark was observed. The paste was then transferred into a plastic silicon mold and cured at ambient temperature into a solid rectangular soap bar.

Determination of the Physicochemical Properties of Soap pH

A 5g sample of the soap shavings was weighed and dissolved in distilled water in a 100cm^3 volumetric flask. The electrode of a standardized pH meter was inserted into the soap solution, and pH readings were recorded. (Warra *et al*., 2012)

Moisture content

5g of soap samples was accurately weighed using an analytical balance of sensitivity 0.1 mg into dried

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tarred moisture dish in an oven for 2hrs at a temperature of 101C. It was allowed to cool and then weighed. The percentage moisture was calculated. (AOAC, 2006)

$$\% \text{ moisture content} = \frac{Cs-C1}{Cs-Cw} x\ 100 \quad (6)$$

C_l = Weight of crucible + sample after floating

Cs = Weight of crucible + sample

C_w = Weight of crucible

Total Fatty Matter (TFM)

The total fatty matter content was determined by reacting soap with the acid in the presence of hot water and measuring the fatty acid obtained. Approximately 10g of the finished soap was weighed and placed in a 250cm^3 beaker, 100cm^3 of distilled water was added, and the mixture was heated in a water bath until the soap melted. Subsequently, 10cm^3 of 20% H_2SO_4 was added, and the mixture was heated until a clear solution was obtained. The fatty acids on the surface of the resulting solution were solidified by adding 5g of candlewax and reheated until the wax melted (Roila *et al*., 2001). The contents were allowed to cool to room temperature to form a cake. The cake was removed, blotted to dry, and weighed to obtain the total fatty matter using the following equation. (Eke *et al*.,2004)

$$\%TFM = \frac{A-M}{W} * 100 \quad (7)$$

A= Weight of wax + Oil

M= Weight of wax

W= Weight of Soap

Free Caustic Alkali (FCA)

A sample of scrapped soap (10g) was placed in a conical flask, and 100cm^3 of neutralized alcohol was added. The flask and the contents were placed in a water bath and heated until the soap dissolved. 10 cm3 of 10% barium chloride solution and 2–3 drops of phenolphthalein indicator were added. The whole content was titrated against 0.1N H_2SO_4 until the solution became colourless. The free caustic alkali was then calculated. (AOAC, 2006)

$$FCA = \frac{0.31}{W} * Va \quad (8)$$

V_A= Volume of acid

W = Weight of Soap

RESULTS

Physicochemical Properties of the oils and their blends

The results obtained for the physicochemical properties of the oil and their blend are presented in Table 2.

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Table 2: Physico-chemical Properties of Palm Oil-Walnut Oil Blend

Samples Value	Blend Comp (P: W)	FFA (mg KOH/g)	Peroxide Value (Meq KOH/g)	Saponification Value (mg KOH/g)	Iodine Value (gI_2 100/g)	Acid (mgKOH/g)
$PW_0 \pm 0.12$	100:0	1.68 ± 0.12	5.0 ± 0.25	234 ± 0.30	55.6 ± 0.25	3.37
$PW_I \pm 0.10$	75:25	1.40 ± 0.10	7.50 ± 0.10	217 ± 0.50	52.0 ± 0.55	2.80
$PW_2 \pm 0.20$	50:50	1.20 ± 0.20	9.09 ± 0.10	199 ± 0.40	46.9 ± 0.60	2.40
$PW_3 \pm 0.10$	25:75	0.95 ± 0.10	11.36 ±0.20	187 ± 0.20	42.4 ± 0.60	1.90
$WP_0 \pm 0.20$	0:100	0.80 ± 0.20	12.5 ± 0.1	182 ± 0.20	37.5 ± 0.50	1.60

P – Palm Oil, W – Walnut Oil, FFA – Free Fatty Acid, Comp – Composition

All values are mean and standard deviation of the samples. Result represent average of two replications.

GC-MS Results and Suggested Compounds

The Gas Chromatographic and Mass Spectra results of the oil blend samples showing some of the suggested compounds are presented in Tables 3 – 5 together with their spectrum. GC-MS analysis was carried out only on the oil blends PW_1 (75:25), PW_2 (50:50), and PW_3 (25:75), and the results obtained were compared with GC-MS results of palm oil and walnut oil in the literature (Ogunmoyela and Ojo, 2020; Rubalya *et al*., 2014).

Table 3 Suggested Compounds in blended Oil PW_1 (75:25)

Peak#	Suggested Compounds	RT	% Area	MF	MW
1	**Tridecanoic acid**	**15.25**	**0.68**	**$C_{13}H_{26}O_2$**	**214.34**
2	**Undecanoic acid**	**16.06**	**1.35**	**$C_{11}H_{22}O_2$**	**186.29**
3	**Cyclooctene, 3-ethenyl-**	**18.68**	**7.18**	**$C_{10}H_{16}$**	**136.23**
4	**Butyraldehyde, 4 (methylenecyclopropyl)-**	**18.84**	**15.49**	**$C_8H_{12}O$**	**124.18**
5	**9,12,15-Octadecatrien-1-ol, (Z,Z,Z)-**	**19.28**	**49.48**	**$C_{18}H_{32}O$**	**264.4**
6	**9,12,15-Octadecatrien-1-ol, (Z,Z,Z)-**	**19.43**	**18.64**	**$C_{18}H_{32}O$**	**264.4**
7	**cis,cis,cis-7,10,13-Hexadecatrienal**	**19.99**	**2.00**	**$C_{16}H_{26}O$**	**234.38**
8	**1,4,9-Decatriene, (E)-**	**31.34**	**3.23**	**$C_{10}H_{16}$**	**136.23**
9	**1,4,9-Decatriene, (E)-**	**32.65**	**1.96**	**$C_{10}H_{16}$**	**136.23**

RT-Retention Time; MF- Molecular formula; MW-Molecular weight

Table 4. Suggested Compounds in Blended Oils of PW_2 (50:50)

Peak#	Suggested Compounds	RT	%Area	MF	MW
1	**Nonanoic acid (Pelargonic)**	**15.06**	**3.66**	**$C_9H_{18}O_2$**	**158.24**

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2	**Cyclohexanebutanoic acid**	**16.49**	**11.57**	**$C_{10}H_{18}O_2$**	**170.25**
3	**10-Undecyn-1-ol**	**18.21**	**13.51**	**$C_{11}H_{20}O$**	**168.28**
4	**10-Undecyn-1-ol**	**18.58**	**25.61**	**$C_{11}H_{20}O$**	**168.28**
5	**11-(2-Cyclopenten-1-yl) undecanoic acid, (+)-(Hydnocarpic acid)**	**19.73**	**10.30**	**$C_{16}H_{28}O$**	**252.39**
6	**2,4-Hexadiene, 1,6-dimethoxy-, (E,E)-**	**22.75**	**5.65**	**$C_8H_{14}O$**	**142.20**
7	**Cyclopropanecarboxylic acid, dodec-9-ynyl ester**	**32.10**	**11.87**	**$C_{17}H_{28}O_2$**	**264.4**
8	**1,5-Heptadiene, (Z)-**	**32.81**	**10.29**	**C_7H_{12}**	**96.17**
9	**10-Undecyn-1-ol**	**34.40**	**7.54**	**$C_{11}H_{20}O$**	**168.28**

RT- Retention Time; MF- Molecular formula; MW-Molecular weight

Table 5: Suggested compounds in Blended Oils PW_3 (25:75)

Peak#	Suggested Compounds	RT	%Area	MF	MW
1	**Undecanoic acid, 10-bromo-**	**15.36**	**2.22**	**$C_{11}H_{21}BrO_2$**	**265.19**
2	**Ethyl .alpha.-d-glucopyranoside**	**17.72**	**10.61**	**$C_8H_{16}O_6$**	**208.21**
3	**9,12-Octadecadienoyl chloride, (Z,Z)-**	**18.36**	**9.00**	**$C_{18}H_{31}ClO$**	**298.9**
4	**10-Undecyn-1-ol**	**18.77**	**23.44**	**$C_{11}H_{20}O$**	**168.28**
5	**10-Undecyn-1-ol**	**18.97**	**10.43**	**$C_{11}H_{20}O$**	**168.28**
6	**1,6-Octadiene**	**19.38**	**11.99**	**C_8H_{14}**	**110.20**
7	**1,4,9-Decatriene, (E)-**	**19.71**	**7.56**	**$C_{10}H_{16}$**	**136.23**
8	**1-(2-Propenyl)cyclopentene**	**20.85**	**7.50**	**C_8H_{12}**	**108.18**
9	**Cycloheptane, bromo-**	**29.79**	**3.10**	**$C_7H_{13}Br$**	**177.08**
10	**Cyclopentaneundecanoic acid**	**36.04**	**8.96**	**$C_{16}H_{30}O_2$**	**254.41**
11	**3-Octen-1-ol, (Z)-**	**36.26**	**5.27**	**$C_8H_{16}O$**	**128.21**

RT- Retention Time; MF- Molecular formula; MW-Molecular weight

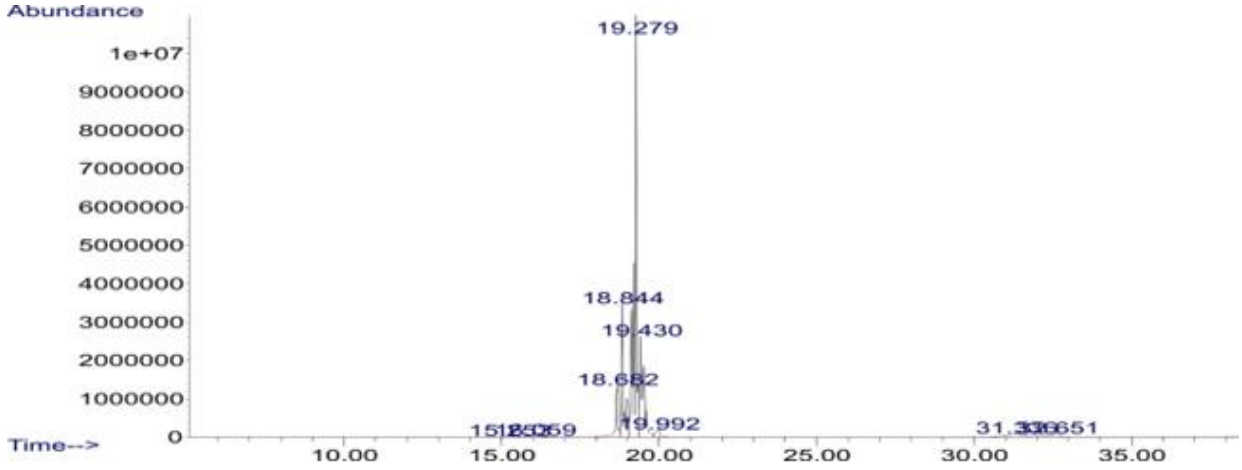

Fig 1. Chromatogram of GC-MS Analysis of Blended Oil PW_1

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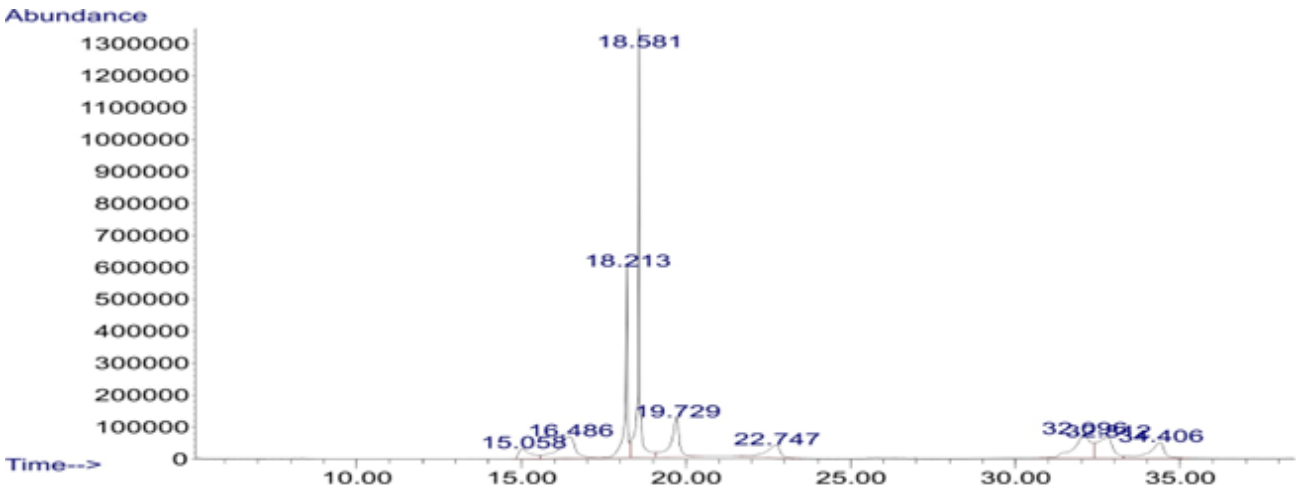

Fig 2. Chromatogram of GC-MS Analysis of Blended Oil PW_2

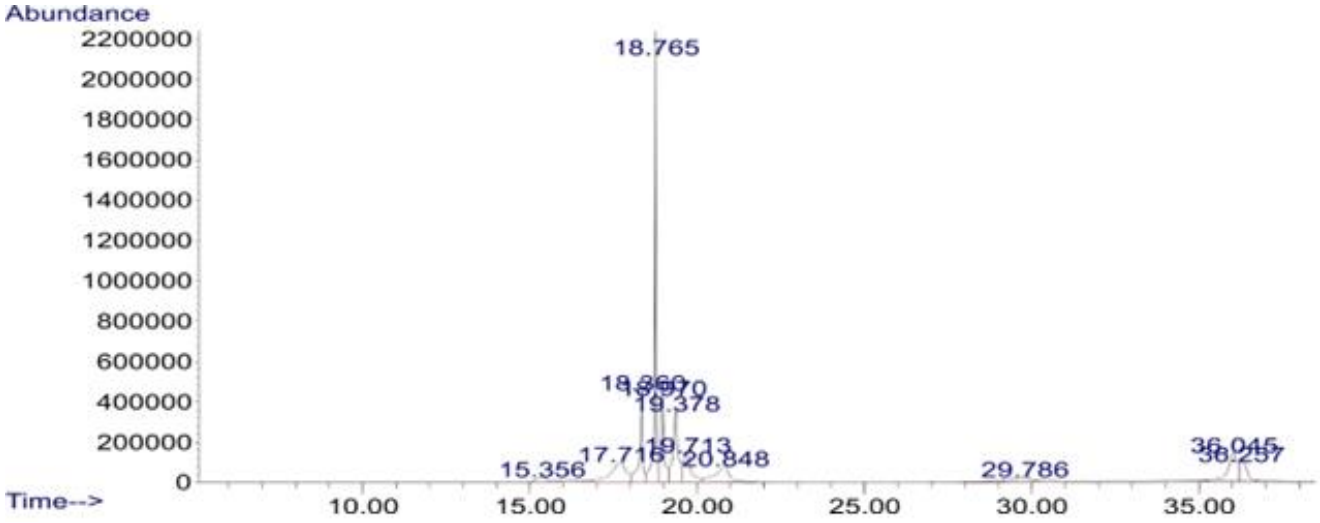

Fig 3. Chromatogram of GC-MS Analysis of Blended Oil PW_3

Physicochemical properties of the Soap Blends

The Physicochemical properties of the soap blend produced from the oil samples are presented in Table 6. Percent moisture content, pH, foam ability, percent total fatty matter and free caustic alkali were the major physicochemical properties deduced.

Table 6 Physico-Chemical properties of the soap blend

Samples	Blend Composition (P:W)	pH	TFM (%)	FCA (g)	Moisture Content (%)	Foam AbilityTest (cm^3)
PW_0	100:0	10.0 ± 0.01	77 ± 0.05	0.79 ± 0.15	12 ± 0.01	150 ± 0.1
PW_1	75:25	10.90 ± 0.02	74 ± 0.1	0.96 ± 0.3	10 ±0.05	126 ± 0.15
PW_2	50:50	12.20 ± 0.03	67 ± 0.15	1.06 ± 0.1	8 ± 0.02	70 ± 0.2

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PW_3	25:75	10.39 ± 0.01	55 ± 0.05	0.93 ± 0.1	12 ± 0.01	68 ± 0.1
WP_0	0:100	10.28 ± 0.01	52 ± 0.1	1.15 ± 0.05	8 ± 0.01	80 ± 0.1

Values are expressed as mean and ± standard deviation of double determinations. P – Palm Oil, W – Walnut Oil, wt – weight, TFM – Total Fatty Matter, FCA – Free Caustic Alkali

DISCUSSION

Oil Yield

The oil yield from African walnut seeds was 40%. Comparing the value with other seeds oil such as corn oil (4.5%), soya beans (21%), Cotton seed (22.9%) as reported by Rahib *et al*., (2015). This shows that African walnuts can produce similar quantities of chestnut and watermelon oil. This also indicates that the processing of African walnuts seed oil for industrial or edible purposes would be economical.

Saponification Value

As shown in Table 2, the saponification value (mgKOH/g) for the PW_0, PW_1, PW_2, PW_3, and WP_0 blends ranged between 182 ± 0.20 – 234 ± 0.30 mgKOH/g. These values are lower than the Avogadro seed oil of 246.7 mgKOH/g (Rahib *et al*., 2015), but greater than that of Moringal Oil, which has a saponification value of 155.8 mgKOH/g (Afolayan *et al*., 2014). Table 4 shows that PW_0 has a higher saponification value, whereas WP_0 has a lower saponification value. This implies that the higher the saponification value, the shorter the fatty acid chain and the lower the molecular weight of the oil and vice versa. On the other hand, the larger the SV, the better the soap-making ability of the oil.

Peroxide Value

The peroxide Value is a measure of the oil deterioration. It gives the extent to which rancidity reactions have occurred during storage. As shown in Table 2, the peroxide values of PW_0, PW_1, PW_2, PW_3, and WP_0 ranged from 5.0 ± 0.25 – 12.5 ± 0.1 Meq/KOH/g. This implies that PW_0 had the lowest peroxide value, whereas WP_0 had the highest. A peroxide value of less than 10 Meq/KOH/g is considered free from rancidity (Obanla *et al*., 2018). PW_0, PW_1, PW_2 blend values are in the range 5.0 ± 0.25 to 9.09 ± 0.10 Meq/KOH/g are considered to be free from rancidity, while PW_3 and WP_0 were 11.36 ± 0.20 and 12.5 ± 0.1 Meq/KOH/g are considered to be rancid.

Iodine Value

The Iodine Value is a measure of the degree of unsaturated fatty acids in an oil and can be used to quantify the number of double bonds present in the oil, which reflects the susceptibility of oil to oxidation (Afolayan *et al*., 2014). As shown in Table 2, the iodine values for samples PW_0, PW_1, PW_2, PW_3, and WP_0 ranged from 37.5 ± 0.50 – 55.6 ± 0.25 gI2100/g. This implies that PW0 has the highest value of iodine and is the most unsaturated, whereas WP0 has the lowest value of iodine and is the least unsaturated. The iodine values of all the samples in Table 2 are lower than the iodine value of water melon seed oil with a value of 121.51 gI2100/g (Duduyemi *et al*., 2013), but greater than that of moringa seed oil with value 35.85 gI2 100/g (Afolyan *et al*., 2014).

Acid Value and Free Fatty Acid

The acidity of oil results from the breakdown of triacylglycerol due to a chemical reaction called hydrolysis, in which free fatty acids are formed. Thus, free fatty acids are a direct measure of oil quality (Hesham *et al*.,

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2015). As shown in Table 2, the free fatty acid values for PW_0, PW_1, PW_2, PW_3, and WP_0 ranged from 1.68 ± 0.12 – 0.80 ± 0.20 mgKOH/g. This implies that PW_0 had the highest value of free fatty acid and WP_0 had the lowest value. Comparing these values with other seed oil, the free fatty acid of PW_3 (0.95) and WP_0 (0.80) has lower values as that of sunflower (0.4); soya beans (0.5), and cotton seed (0.7) while PW_0 (1.68), PW_1 (1.40) and PW_2 (1.20) have higher values as that of corn oil (1.5) and chestnut (3.01) as reported by Rahib *et al*., (2015).

GC-MS interpretation of the Oil-Blend

The GC-MS results of the PW_1 oil blend in Table 3 show that there are nine (9) compounds present in the oil blend, two of which are saturated fatty acids, tridecanoic acid, and undecanoic acid. In the PW_2 oil blend, GC-MS results in Table 4 show that there are nine compounds present in the oil blend, two of which are fatty acids: nonanoic acid (a saturated fatty acid) and hydnocarpic acid (a monounsaturated fatty acid). In the PW_3 oil blend, GC-MS results in Table 5 show that there are 11 compounds present in the oil blend, one of which is a saturated fatty acid, 10-bromoundecanoic acid. Comparing the GC-MS results of the oil-blend PW_1, PW_2 and PW_3 in table 3.- 5 with GCMS results of the individual oils in literature review, it was shown that all the fatty acid compound found in the oil-blends were present in the individual oils.

Quality Parameter of the Soap Samples produced

Moisture Content

Moisture content is used to assess the shelf life of a product. (Idoko *et al*., 2018). As shown in Table 6, the moisture contents of PW_0, PW_1, PW_2, PW_3, and WP_0 ranged from 8.0 ± 0.01% to 12 ± 0.01%. PW_0 had the highest moisture content, whereas PW_3 and WP_0 had the lowest. The % moisture content of commercial bar soap lux (16.65) and premier (15.94), as reported by James (2012), was higher than the moisture content of all the soap samples produced in Table 6. However, the moisture contents of PW_0, PW_1, and PW_3 fell within the limits (10 – 15%) of the Encyclopedia of Industries Chemical analysis, while PW_2 and WP_0 did not.

pH of the Soap Samples

As shown in Table 6, the pH values of the soap samples produced PW_0, PW_1, PW_2, PW_3, and WP_0 ranged between 10.28 ± 0.01 – 12.20 ± 0.03. PW_2 had the highest pH, whereas WP_0 had the lowest value. However, the pH value of the PW_0, PW_1, PW_3, and WP_0 falls within the acceptable limits of pH (9-11) in soaps set by Standard Organization of Nigeria while PW_2 do not, this was as a result of incomplete hydrolysis resulted from saponification process (Idoko *et al*., 2018)

Foam Ability Test

The foam Ability Test of soap (or the measure of foam height) is an important factor that attracts consumer interest, even though it has little contribution to the cleansing ability of the soap (Mak-Mensah and firepong, 2011).

As shown in Table 6, the foaming ability of the soap samples PW_0, PW_1, PW_2, PW_3, and WP_0 ranged from 68 ± 0.1 – 150 ± 0.1. PW_0 had the highest value of foaming ability while PW_3 had the lowest.

Free Caustic Alkali Content of the Soap Samples

The Free Caustic Alkali of soap is a measure of its abrasiveness or roughness. (Ashrafy *et al*., 2016) As shown in Table 6, the free caustic alkalis of PW_0, PW_1, PW_2, PW_3, and WP_0 ranged from 0.93 ± 0.1 – 1.15 ± 0.05g. WP_0 had the highest FCA, whereas PW_3 had the lowest. However, the free caustic alkalis of PW_0, PW_1, PW_2, PW_3, and WP_0 were higher than the acceptable limits set by the Standard Organization of

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Nigeria (≤ 0.05). The excess free caustic alkali present in all of the soap samples produced can have adverse effect on the skin or cloth but it can be reduced by adding humectants such as propylene glycol, glycerol on the soap (Woollatt, E. 1985).

Percent Total Fatty Matter

Table 8 shows that the % TFM of the soap samples ranged from 52 ± 0.1 – 77 ± 0.05%. PW_0 had the highest value, whereas WP_0 had the lowest value. The TFM of PW_0 (77%) and PW_1 (74%) falls within the limits set by the International Standard Organization (ISO 685) which is 73-77%, indicating good quality soap. PW_2, PW_3, and WP_0 fall below the limits set by ISO because of the presence of unreacted NaOH in the soap of PW_2, PW_3, and WP_0.

CONCLUSION

Quality parameters such as total fatty matter, free alkali, moisture content, pH, and foam ability were investigated for the bar soaps produced. The total fatty matter of PW_0 and PW_1 soaps is said to fall within the acceptable limits set by the International Standard Organization, while PW_2, PW_3, and WP_0 fall below the limit set by the ISO. The pH values of all the bar soaps produced are within the limit set by the Standard Organization of Nigeria (9-11) except for PW_2 soap, which is 12.20. Thus, from the results obtained in this study, it can be concluded that because of the favorable physicochemical properties (high % TFM, normal pH value, and high foam ability) of PW_0 and PW_1, these soaps can be recommended for laundry.

RECOMMENDATIONS

From the analysis of the oil and the bar soaps produced;

1. It is recommended that a commercial plant be designed and set up to extract walnut oil from the seed in large quantities for commercial purposes.
2. It is recommended that the waste product from walnut fruit be converted to other by-products such as biofuels.

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