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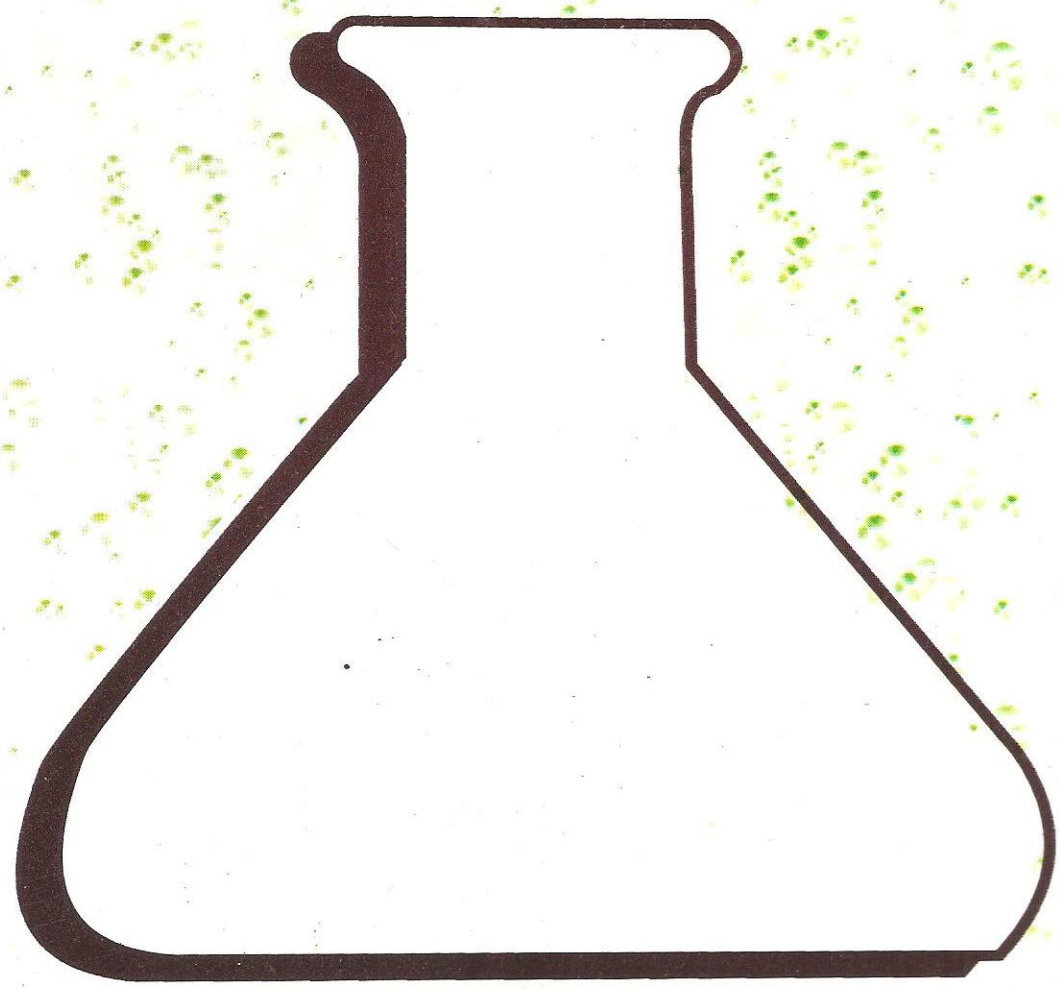

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Comparative Analysis of Physico-Chemical Properties of Extracted and Collected Palm Oil and Tallow

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ABSTRACT

Palm oil was extracted from palm fruits while animal fat (tallow) from Cow. Various samples of both palm oil and tallow were also obtained from the market. Determination of the physico-chemical properties were made on these reference and collected samples. The results obtained from the reference samples of both palm oil and tallow are as follows: Relative density: palm oil; 0.875 and tallow; 0.909. Moisture content: palm oil; 0.24%m/m and tallow; 0.13%m/m. Free fatty acid: palm oil; 2.12% and tallow; 0.559%. Acid value: palm oil; 4.217 mg KOH / g of oil and tallow; 1.113 mg KOH / g of oil. Saponification value: palm oil; 191.324 mg KOH / g of oil and tallow; 190.763 mg KOH / g of oil. Peroxide value: palm oil; Nil and tallow; Nil. Unsaponifiable matter: palm oil; 3.033g/kg and tallow; 1.133g/kg. Iodine value: palm oil; 53.870 and tallow; 44.154. Soap content: palm oil; 0.003m/m and tallow; 0m/m. Insoluble impurities: palm oil; 0.001m/m and tallow; 0.001m/m. Titre value: palm oil; 41.5 ^{o}C and tallow; 42.5 ^{o}C. Rancidity: palm oil; negative and tallow; negative. The conclusion derived from these analyses is that, some of the samples do not comply with the standards given by the regulatory bodies (Standard Organization of Nigeria, and National Food and Drug Administration and Control).

Keywords: Free fatty acid, Lipids, Palm oil, Rancidity, Saponification, Tallow.

Introduction

Lipids are a group of substances that in general, are soluble in ether, chloroform, or other organic solvents but are sparingly soluble in water. However, some lipids, such as triacylglycerols, are very hydrophobic. The term fat signifies extracted lipids that are solid at room temperature and oil refers to those that are liquid at room temperature. However, the three terms lipid, fat and oil are often used interchangeable (Nielsen, 2002). In plants, lipids are of two types; the structural and the storage. The former are present as constituent of various membranes and protective surface layers and make up about 7% of the leaves of higher plant (Umar, 2005), while the latter occurs in fruits and seeds and are predominantly oil (McDonald *et al*., 1995). Fats and oils are characterized by a selection of chemical and physical test methods. One of the most important tests is the determination of the fatty acid (FA) composition, which is done via Gas chromatograph (GC) (Craske *et al*., 1988). The chemical properties of fats and fatty acids may be expressed in terms of the reactions of the carboxyl groups and the hydrocarbon chain attached to it, which may be saturated or unsaturated. Hydrolysis, alcoholysis, autoxidation, and sulphurnation, are some of the possible reactions that fats and fatty acids can undergo.

Palm oil is composed of fatty acids, esterifies with glycerol just like any ordinary fat. Fatty acids are saturated and unsaturated aliphatic carboxylic acids with carbon chain length in the range of C_6 up to C_{24} e.g. CH_3-$(CH_2)_{14}$-COOH. It is high in fatty acid, about 50%. The oil palm gives its name to the 16 carbon saturated fatty acid palmitic acid found in palm oil. Monounsaturated oleic acid is also a constituent of palm oil. Palm oil is the largest source of tocotrienol; part of the vitamin E family and it is also high in vitamin K and dietary magnesium. The split-off fatty acids are a mixture of fatty acids ranging from C_6 to C_{18} depending on the type of oil or fat (Faessler, 2004). As palm oil is rich in carotene, it can be used in place of cod liver oil for correcting vitamin A deficiency. Each 100g of the fruit contains 540 calories, 26.2g H_2O, 1.9g protein, 58.4g fat, 12.5g total carbohydrates, 3.2g fiber, 1g ash, 82mg Ca, 47mg P, 4.5mg Fe, 42.42mg β-carotene equivalent, 0.2mg thiamine, 0.1ng riboflavin, 1.4mg niacin, and 12mg ascorbic acid (Barker and Worgan, 1981). The oil contains, per 100g, 878 calories, 0.5% H_2O, 0% protein, 99.1% fat, 0.4g total carbohydrates, 7ng Ca, 8mg P, 5.5mg Fe, 27.28mg β-carotene equivalent, 0.03mg riboflavin, and a trace of thiamine. The glycerides composition of the oil are; 45% oleodipalmitins, 30%

palmitodioleins, 10% oleopalmatostearins, 6-8% linoleodioleins, and 6-8% fully saturated glycerides, tripalmatin and diapalmitostearin (Barker and Worgan, 1981).

Tallow (animal fat) is rendered from beef or mutton fat, processed from suet (the fatty tissue about the loins and kidneys of sheep, oxen etc.) (Appleby and Halloran, 1990). By definition, tallow is animal fat with "titre" of greater than 40^{o}C. This means that upon cooling a melted fat sample, the temperature at which it re-solidifies is 40^{o}C or greater. Thus, tallow is solid or semi-solid at room temperature. There is a preponderance of data that supports a greater consumption of poly-unsaturated fats like the essential fatty acids linoleic acid (LA) and the omega-3 fatty acids like linolenic (ALA), eicosapentaenoic (EPA) and docosahexaenoic (DHA) acid by dogs and cats for the management of inflammatory diseases and neurological development (Aldrich, 2007).

The fatty acids in beef tallow are about 50% saturated, with a small amount of LA (3.0%) and ALA (0.6%) and none of the longer chain omega-3s (EPA or DHA). Mutton tallow has a similar level slightly higher level of LA (5.5%) and ALA (2.3%). In general, tallow is good "platform" to provide energy and flavor, but a balanced diet may require an enrich meat with LA and (or) omega-3 fatty acids (Aldrich, 2007).

It has been observed that most of the oils and fats in the market are adulterated in one way or the other, through the use of unripe fruits or the use of tallow already affected with bacteria or the use of substandard processing methods or the combination of different oil plant fruits or animal fats as the case may be. This tends to cause a great detriment to the health of the consumers of the lipids products. The significance of the research is that, the findings could be used in deciding quality of lipid samples from different parts of the country; their level of adulteration and proffer solutions to ways of checkmating the contamination. Therefore it is within the aim of this research to determine the physico-chemical properties such as; relative density, moisture contents, free fatty acids, acid values, saponification values, unsaponifiable matter, soap contents, insoluble impurities, iodine values, peroxide values, rancidity and titre values of both extracted (reference samples) and the collected samples of the palm oil and tallow, and compare the results of the reference and mean of the collected samples with that of the known.

Materials and Method

The methods used for the analysis of the physico-chemical parameters in this research work, are the traditional basic method which are the official methods approved by Standard Analytical Chemistry bodies (Nielsen, 2002; A.O.A.C, 1990; A.O.C.S, 1990; I.U.P.A.C, 1987; Milwidsky and Gabriel, 1982 and S.O.N, 2000).

Sampling and Sampling Pre-treatment

Thirteen different samples of palm oil were analyzed with reference sample extracted in the laboratory, while others were collected from different part of the country (Table 1) as finished products. The palm fruit (used as the reference sample) was collected from Umutu in Delta State, Nigeria. The palm fruits were washed with ordinary water (to free them from soil and other particles). Four different tallow samples were analyzed, namely: edible tallow (reference sample), top white tallow, bleached fancy tallow and top white grease with the reference sample sourced locally while others were imported (Table 2 below). The animal (Cow) fat (used as reference sample) was collected from abattoir in Kano metropolis, Kano State, Nigeria. Each sample was washed with distilled water to free it from blood and other particles such as lean meat.

Table 1: Sample Source (Palm Oil)

SL	Sample Source
A	Reference sample
B	Calabar
C	Calabar
D	Agbor
E	Ibadan
F	Okene
G	Okumu
H	Okumu
I	Owerri
J	Sapele
K	Bidda
L	Aba
M	Owerri

Table 2: Sample Source (Tallow)

SL	Sample Source
A	Edible tallow (Reference sample)
B	Top white tallow (New Zealand)
C	Bleached fancy tallow (New Zealand)
D	Top white grease (New Zealand)

Palm oil / Tallow Extraction methods

Both the palm oil and tallow used as reference samples were extracted using the traditional rendering method. The fatty tissue was heated with steam or hot water to melt followed by the separation of the oil or fat from the aqueous layer.

The fruits from the palm sample was boiled for one hour with water, drained and then crushed with mortar and pestle (to separate the mesocarp from the kernel). Hot water was then used to extract the oily juice from the chaff and kernel by boiling the oil juice until the oil settled at the surface. The oil was scooped at intervals until there was no more oil on the surface of the boiling juice.

While the tallow sample was extracted by cutting the cleaned fat into pieces and placing them in a pot containing some water. The pot content was then boiled over a low to medium heat, stirring gradually at intervals (to avoid sticking to the bottom of the pot). As the water boiled off; the molten fat was scooped at intervals, until all the fat melt into liquid leaving some small particles (cracklings). It was then allowed to cool for a while (Atkins, 2007).

Determination of Some Physico-Chemical Parameters of Palm oil and Tallow.

a. Relative Density (RD)

A cleaned, dried and pre-weighed density bottle was filled with distilled water to the mark ($50cm^3$) and maintained in a water-bath until the temperature of the water reached 20^oC. The bottle outside was wiped and weighed (m1). The same bottle was then emptied and dried and then filled with the sample to the same mark ($50cm^3$), then maintained in the water-bath until the temperature of the oil sample inside reached 40^oC. The bottle outside was wiped and weighed (m2). The process was done in triplicate for all the palm oil and tallow samples. The relative densities of the samples were then calculated using equation (S.O.N, 2000; Milwidsky and Gabriel, 1982)

$$\text{RD} = \frac{m2}{m1}[1 + x(t - 20)°\text{C}]$$

Where: RD = Relative density, m1 = mass of water, m2 = mass of fat or oil, $t = 40^oC$
And x = Coefficient of cubical expansion for borosilicate glass (0.00001) K^{-1}

b. Moisture Content (MC)

The moisture content palm oil and tallow was each determined using the Mettler Toledo Moisturizer (HB43 Halogen); it's a computerized machine that consists of a scale, oven, time and a print-out screen. The moisturizer was set to 105^oC (378K) in 30minutes. To a tarred aluminum dish in the moisturizer; weighed sample (7.0g) was placed and the machine closed to start automatically. After 30minutes, the result was read-off the print-out screen as it appears in percentage (S.O.N, 2000).

c. **Free Fatty Acid (FFA)**

The sample (10.0g) of palm oil / tallow was weighed into $250cm^3$ boiling flask, followed by the addition of neutralized alcohol (ethanol, $150cm^3$). The mixture was boiled on a hot plate until all the oil dissolved completely and then phenolphthalein (3-4drops) was added. The solution was titrated with 0.1M sodium hydroxide until a faint pink end point was observed and the titre value (T) recorded. The percentages FFA of the samples were calculated using equation:-

$$\%\text{FFA (as oleic)} = \frac{(T \times M \times 28.2)}{W}$$

Where: %FFA = Percentage free fatty acid, T = Volume of Sodium hydroxide used,
M = Molarity of Sodium hydroxide used, W = Weight of sample used.
28.2 = constant use for calculating Oleic. (S.O.N, 2000; Milwidsky and Gabriel, 1982)

d. Acid Value (AV)

The same procedure was repeated as that of the free fatty acid, but the equation used for calculation is as presented below. (Milwidsky and Gabriel, 1982)

$$\text{AV} = \frac{(56.1 \times M \times T)}{W}$$

Where: AV = Acid value, T = Volume of Sodium hydroxide used, M = Molarity of Sodium hydroxide used, W = Weight of sample used, 56.1 = Molecular weight of Potassium hydroxide.

e. Saponification Value (SV)

The sample (4.0g) of either palm oil / tallow was weighed into an Erlenmayer flask and to this was added 0.5M alcoholic KOH (50cm^3). The mixture was then heated to saponify the fat or oil. The unreacted KOH was then back titrated with 0.5M HCl using phenolphthalein as indicator. A blank sample was also prepared and back titrated accordingly. The sample and blank titres (V1 and V2) were recorded. The saponification values of the samples were then calculated using equation. (Nielsen, 2002; Milwidsky and Gabriel, 1982)

$$\text{SV} = \frac{[(V2-V1)\ x\ M\ x\ 56.1]}{W}$$

Where: SV=Saponification value, V1=Volume of Hydrochloric acid used for the sample,
V2 = Volume of Hydrochloric acid used for the blank,
M = Molarity of the Hydrochloric acid, W = Weight of sample used,
56.1 = Molecular weight of Potassium hydroxide.

f. Unsaponifiable Matter (USM)

The sample of palm oil / tallow (4.0g) was weighed into an erlmayer flask and to this was added 0.5M alcoholic KOH (50cm^3). The mixture was then heated to saponify the fat or oil. The unreacted KOH was then back titrated with 0.5M HCl using phenolphthalein as indicator. The neutralized solution above, which contains the soap formed was then transferred into a separatory funnel, with repeated washing (thrice) using about 50% aqueous alcohol (10cm^3). The Unsaponifiable matters were then extracted with petroleum ether (50cm^3) thrice consecutively. The three petroleum ether extracts were then combined and washed with water: neutralized ethanol (1: 1, 50cm^3) mixture and the washings were discarded. The extracts were then filtered through filter paper into a dried tarred flask (W1), rinsing the separatory funnel and the filter paper with petroleum ether (10cm^3). The filtrate was evaporated to dryness on a water-bath, to which acetone (10cm^3) was added when it was about to dry (to remove traces of water) and evaporates them off.The flask was dried in an oven to constant weight and re-weighed (W2). The unsaponifiable matters of the samples were then calculated using equation (Nielsen, 2002; Milwidsky and Gabriel, 1982).

$$\%\text{USM} = 100\ \frac{(W2-W1)}{M}$$

Where: %USM = Percentage Unsaponifiable matter, M = Mass of sample used, W1 = Weight of flask without extract, W2 = Weight of flask with extract.

g. Iodine Value (IV)

Palm oil / tallow sample (0.2g) was weighed into a conical flask and carbon tetrachloride (10cm^3) was then added to dissolve it, followed by Wij's solution (20cm^3) was added and stoppered immediately then swirled. The mixture was allowed to stand in the dark at ambient temperature for 30min. Halogen addition to double bond takes place. A 15cm^3 solution of 10% KI was then added with water (100cm^3) to rinse the flask. This is to reduce excess ICl_3 and free iodine. The solution was then titrated (librating the iodine) with a solution of sodium thiosulphate (0.1M) using starch as an indicator. A blank sample was also prepared and back titrated accordingly.

The sample results (V1) and that of the blank (V2) were recorded. The Iodine values of the samples were then calculated using the equation (Milwidsky and Gabriel, 1982; A.O.A.C, 1990; Nielsen, 2002).

$$\text{IV} = \frac{[(V2-V1) \times M \times 12.7]}{W}$$

Where: IV = Iodine value, V1 = Volume of Sodium thiosulphate used for the sample.
V2 = Volume of Sodium thiosulphate used for the blank,
M = Molarity of Sodium thiosulphate used, W = Weight of sample used.
12.7 = ~ constant used to convert from milliequivalent thiosulphate to gram (Molecular weight of Iodine = 126.9)

h. Soap Content (SC)

A glass stoppered test tube was rinsed with the test solution (0.5cm^3 bromophenol blue in 100cm^3 aqueous acetone titrated to just yellow colour with 0.01M HCl), and a weighed palm oil / tallow sample (40.0g) was added and shaken with water (10cm^3). The solution was shaken and warmed on a water-bath. To the resulting solution, acetone (50cm^3) was added, shaken vigorously and allowed to stand until two layers separated out. A blue-green colouration in the upper layer indicated the presence of soap, otherwise, no need to titrate.

The blue-green solution was titrated with 0.01M HCl with a 25cm^3 micro-burette until the yellow colour is restored. The soap content as sodium oleate of the samples was calculated using equation (A.O.A.C, 1990; A.O.C.S, 1990; Milwidsky and Gabriel, 1982).

$$SC = \frac{(0.301 \times T)}{M}$$

Where: SC = Soap content, T = Volume of Hydrochloric acid used, M = Mass of sample used.

i. Insoluble Matter (IM)

The palm oil / tallow sample (20.0g) was weighed into a flask and melted. Petroleum ether (20cm3) was then added and the flask was closed immediately; shaken vigorously and allowed to stand for 30minutes. The content was then filtered through a filter paper (whatman) which has been dried and weighed (W1). The residue was carefully washed with small amount of petroleum ether, until the filter paper was free of oil and then dried with the filter paper then re-weighed (W2). The Insoluble matter of the samples was calculated using equation (A.O.A.C, 1990; A.O.C.S, 1990; Milwidsky and Gabriel, 1982).

$$\%IM = \frac{(W2 - W1)}{(10\ MS)}$$

Where: %IM = Percentage Insoluble matter, MS= Weight of sample used,
W1=Weight of residue without filter paper, W2 = Weight of residue with filter paper.

j. Titre Value (TV)

To the sample (50.0g) weighed into the conical flask was added sodium hydroxide (15.0g) and ethanol (100cm^3). The mixture was then saponified (turned to soap) and the soap formed transferred into a 1dm^3 beaker while still hot and stirred to dissolve with hot water (400cm^3). This solution was then acidified with 3M HCl, while methyl orange indicator (3-4drops) was added until the indicator colour changed to distinct red colour. The hot molten fatty acid was then filtered through a dry coarse filter paper (to absorb any traces of water), poured into a clamped test tube and allowed to cool to 50^{o}C. A 0-50^{o}C thermometer graduated in 0.2^{o}C was then lowered into the center of the molten fatty acid, stirred rapidly after some second and the thermometer returned back to its position. The highest temperature attained when the temperature fall slightly and rises before falling again is the titre value (I.U.P.A.C, 1987; A.O.A.C, 1990; A.O.C.S, 1990).

k. Peroxide Value (PV)

To a weighed sample (1.0g) in a flask, was added powdered potassium iodide (1.0g) and solvent mixture (2: 1, glacial acetic acid: chloroform v/v). The resulting solution was then placed on a water-bath to dissolve properly and 5%potassium iodide (20cm^3) was then added. The sample solution was then titrated with 0.002N sodium thiosulphate using starch as indicator.

The peroxide values of the samples were calculated using equation (Nielsen, 2002; I.U.P.A.C, 1987).

$$PV = 2 \times V$$

Where: PV = Peroxide value, V = Volume of Sodium thiosulphate used, 2 = (N x 1000) / W,
N = Normality of Sodium thiosulphate used, W = Weight of sample used.

l. Rancidity (R)

The Kreis test for rancidity was used. The sample ($10cm^3$) was melted in a stopperred test tube. 0.1% phloroglucinol solution ($10cm^3$) was then added, followed by concentrated HCl ($10cm^3$). The mixture was shaken vigorously; a pink colour indicates incipient rancidity. The sample results were then recorded as; + (rancid) and – (not rancid), (A.O.A.C, 1990; A.O.C.S, 1990).

Results and Discussion

The results of the analysis of the reference samples, the mean of the collected samples and the standard deviations of the collected samples of both palm oil and tallow are compared in Table 4 and Table 5 respectively, while Table 6 and Table 7 compare results of the reference samples, the mean of collected samples, the standard deviations of the collected samples and their standards.

Though, the results of some collected samples of palm oil are found to be high compared to the reference samples example; MC (% m/m) of sample B =4.69,C=5.14,and D=1.27 and FFA (%) of samples B=7.673, F=8.153, J=7.027,K=7.585 and M=10.77 where samples B,E,F and M are found to be rancid while all the collected samples have lower SV (mg KOH / g of oil) values than the reference sample, except that of sample H =191.114 that is very close. Likewise, same also occur in the case of collected tallow samples where sample D has a high MC (% m/m) value of 1.05 while both samples B and D are also found to be rancid when compared with the tallow reference sample.

Table 3: KEYS

Parameters.	Abbreviations.
Relative density (40^oC /20^oC)	RD
Moisture contest (% m/m)	MC
Free fatty acid (%)	FFA
Acid value (mg KOH / g of oil)	AV
Saponification value (mg KOH / g of oil)	SV
Unsaponifiable matter (g / kg)	USM
Iodine value	IV
Soap content (% m / m)	SC
Insoluble matter (% m/ m)	IM
Titre value (^oC)	TV
Peroxide value (meq / kg)	PV
Rancidity (+ / -)	R
Mean	MN
Sample label	SL
Milliequivalent	meq
Standard deviation	SD

Table 4: Comparison between Palm oil Analysis Results of Physico-chemical Parameters of Reference Sample with the mean and Standard deviation of its Collected Samples.

Parameters	Reference Samples	Mean ± SD of Collected Samples
Relative density (40°C /20°C)	0.875	0.907±0.008
Moisture contest (% m/m)	0.24	1.563±1.600
Free fatty acid (%)	2.120	5.939±2.280
Acid value (mg KOH / g of oil)	4.217	11.818±4.540
Saponification value (mg KOH / g of oil)	191.324	186.934±3.460
Unsaponifiable matter (g / kg)	3.033	5.888±2.060
Iodine value	53.870	57.828±4.690
Soap content (% m / m)	0.003	0.005±0.005
Insoluble matter (% m/ m)	0.001	0.014±0.009
Titre value (°C)	41.500	41.460±0.720
Peroxide value (meq / kg)	0.000	0.801±1.070
Rancidity (+ / -)	-	-

Table 5: Comparison between Tallow Analysis Results of Physico-chemical Parameters of Reference Sample with the Mean and Standard deviation of its Collected Samples.

Parameters	Reference Samples	Mean ± SD of Collected Samples
Relative density (40°C /20°C)	0.909	0.900±0.006
Moisture contest (% m/m)	0.13	0.490±0.490
Free fatty acid (%)	0.559	2.848±0.620
Acid value (mg KOH / g of oil)	1.113	5.666±1.220
Saponification value (mg KOH / g of oil)	190.763	188.106±1.880
Unsaponifiable matter (g / kg)	1.330	4.703±1.150
Iodine value	44.154	42.785±6.220
Soap content (% m / m)	0.000	0.004±0.003
Insoluble matter (% m/ m)	0.001	0.004±0.005
Titre value (°C)	42.500	41.500±0.500
Peroxide value (meq / kg)	0.000	2.400±1.350
Rancidity (+ / -)	-	+

Table 6: Comparison between Palm oil Analysis Results of Physico-chemical Parameters of Reference Sample with the Mean and Standard deviation

of the Collected Samples and their Standards (S.O.N., 2000; Milwidsky and Gabriel, 1982)

Parameters	Reference Samples	Mean ± SD of Collected Samples	Standard Values
Relative density (40°C /20°C)	0.875	0.907±0.008	0.898-0.907
Moisture contest (% m/m)	0.240	1.563±1.600	0.200 max
Free fatty acid (%)	2.120	5.939±2.280	Nil
Acid value (mg KOH / g of oil)	4.217	11.818±4.540	Nil
Saponification value (mg KOH / g of oil)	191.324	186.934±3.460	185-205
Unsaponifiable matter (g / kg)	3.033	5.888±2.060	10.000
Iodine value	53.870	57.828±4.690	45-55
Soap content (% m / m)	0.003	0.005±0.005	0.005
Insoluble matter (% m/ m)	0.001	0.014±0.009	0.050
Titre value (°C)	41.500	41.460±0.720	42.000
Peroxide value (meq / kg)	0.000	0.801±1.070	10.000
Rancidity (+ / -)	-	-	-

Table 7: Comparison between Tallow Analysis Results of Physico-chemical Parameters of Reference Sample with the Mean and Standard deviation of the Collected Samples and their Standards. (S.O.N., 2000; Milwidsky and Gabriel, 1982)

Parameters	Reference Samples	Mean ± SD of Collected Samples	Standard Values
Relative density (40°C /20°C)	0.909	0.900±0.006	0.893-0.904
Moisture contest (% m/m)	0.13	0.490±0.490	0.300 max.
Free fatty acid (%)	0.559	2.848±0.620	2.000
Acid value (mg KOH / g of oil)	1.113	5.666±1.220	2.500
Saponification value (mg KOH / g of oil)	190.763	188.106±1.880	190-202
Unsaponifiable matter (g / kg)	1.330	4.703±1.150	12.000
Iodine value	44.154	42.785±6.220	32-50
Soap content (% m / m)	0.000	0.004±0.003	0.005
Insoluble matter (% m/ m)	0.001	0.004±0.005	0.050
Titre value (°C)	42.500	41.500±0.500	40-49
Peroxide value (meq / kg)	0.000	2.400±1.350	16.000
Rancidity (+ / -)	-	+	-

To discuss the results obtained on the physiochemical parameters, it would be more appropriate to compare the Reference sample, Mean of the collected samples and the Industrial standards for each parameter. This is to check deviation of either the sample used as reference or the collected market samples from the Standard value for each parameter.

Relative density is used to aid in checking adulteration of the oil or fat sample with impurities like water, sludge or alcohol. In the results obtained, both the mean of the collected palm oil samples and the reference sample are within the range of the standard (0.89-0.9), i.e. 0.907 and 0.875 respectively. This indicates that the palm oil samples were not adulterated with either water or alcohol. This goes along for the relative density of tallow analysed i.e. the collected samples and the reference are also within the range of the standard (0.893-0.9) (S.O.N, 2000).

Moisture content is actually a confirmatory check on the dryness of the oil or fat sample. A palm oil sample has a maximum moisture content of 0.2% m/m. In this vain, considering the results obtained, we can say the collected samples mean moisture content (1.503% m/m) is higher than that of the standard; which might be due to adulteration with water. Even though, the relative density is within the range of

the standard as seen above. This can be confirmed from the result of the moisture content of the reference sample (0.24% m/m), which is in conformity with the standard of 0.2% m/m maximum.

On the other hand, the moisture content of mean of collected tallow samples (0.49%, ±0.490 m/m) tends to deviate a little from the standard of 0.3% m/m maximum. The reference sample and one of the collected sample (sample C) tends to be within the standard range i.e. 0.13% m/m and 0.19% m/m respectively. While another collected sample (sample D), is highly wet with moisture content of 1.05% m/m. This might be due to handling or deliberate adulteration by the provider.

Since fats and oils are triglycerides, the free fatty acids should be very low in highly graded lipid sample. In addition to free fatty acids, acids phosphates and amino acids can also contribute to acidity. Free fatty acids (FFA) is the percentage by weight of a specified fatty acid (e.g. oleic or lauric acid). For fatty acids, the acid value, in conjunction with the saponification value, can be used to give a measure of the amount of neutral fat present. The FFA is normally expressed as oleic acids. The exceptions are coconut and palm kernel oils, which are calculated as lauric acids. Where the FFA is expressed as oleic acids, the acid value, for all the intents and purposes, is double the FFA figure (Milwidsky and Gabriel, 1982).

The standard FFA is NIL for palm oil. In this vain, the results obtained in the analysis showed that the reference sample is having FFA value of 2.120%, which is quite close to the standard when compared with the result obtained for the mean of the collected samples i.e. 5.939%. This indicates a high level of unesterified long chain fatty acids, with one of the samples (sample M) having a very high FFA value of 10.770%, all directing to high deviation, from the standard.

On the other hand, the standard FFA value in tallow is 2% maximum. In the results obtained, the reference sample is having a lower FFA value than the standard i.e. 0.559%, confirming its good quality. While the mean FFA value for the collected samples is higher than that of the standard i.e. 2.8%, as even one of the samples is having a very higher average FFA of 3.55% (sample D).

Acid Value (AV) and Free Fatty Acid (FFA) are analytically used to detect the level of unesterified fatty acid in a lipid sample to define its quality. Both the AV and FFA are used to estimate the amount of oil that will be lost during refining steps designed to remove fatty acid, i.e. in the production of RBDPO (Refined Bleached Deodourized palm oil). Hence, high acidity level means a poorly refined oil or fat breakdown after storage or use. If the fatty acid librated is volatile, AV or FFA may be a measure of hydrolytic rancidity.

The standard Acid value (AV) in palm oil is NIL. Thus, from the results obtained, the AV of 4.2 mg KOH / g of oil for the reference sample and 11.8 mg KOH / g of oil for the mean of the collected samples indicate respective high level of long chain carboxylic acids in the palm oil samples. This result confirmed the results obtained in the initial FFA analysis.

As expected, the AV in the reference sample of the tallow (1.113 mg KOH / g of oil) is lower than that of the standard (2.5 mg KOH / g of oil), while that of the mean of the collected samples is higher than that of the standard. Both results go along with the results obtained from the FFA analysis on the tallow previously reported. Furthermore, sample D, which has highest average FFA of 3.55 mg KOH / g of oil has the highest AV of 7.069 mg KOH / g of oil.

The Saponification value (SV) is used to determine the saponification number of a fat or oil which is an index of the average molecular weight of the triacylglyceride in the sample. Saponification number is a very important factor in soap production. The smaller the SV the higher or longer the average fatty acid chain length. Although, adulteration of fat or oil with unsaponifiable matter can lead to drop in saponification value (Nielsen, 2002). From the results obtained, we can confidently say that both the reference sample and the mean of the collected samples are having SV within the range of the standard i.e. 185-205 mg KOH / g of oil. The results for the SV even indicated that all the collected samples were having lower values than the reference sample, which has SV of 191.3 mg KOH / g of oil. On the other hand, all the collected samples in the tallow have lower SV than the standard of 190-202 mg KOH / g of oil. The mean of the collected samples is 188.106 mg KOH/ g, indicating that they are not that suitable for soap production. The reference sample goes in conformity with the standard range, and has a SV of 190.7 mg KOH / g of oil.

The Unsaponifiable matter (USM) is defined as the oily (petroleum-ether soluble) matter which cannot be converted into soap after saponification. Glycerol and other water soluble alcohols are not determined. The amount of USM found in edible fat and oil are usually small. Hence, high figure may indicate adulteration. As expected, the higher the SV; the lower would be the USM value. Both reference sample and the mean of collected samples' results obtained are lower than the standard value of 10 g / kg. The reference sample has lower USM value of 2.03 g / kg, proportionate to its SV of 191.3 mg KOH / g of oil, which is higher than the mean of the collected samples of 186.96 mg KOH / g of oil, proportionate to its USM value of 5.888 g / kg. This value also indicates and confirmed that, the reference sample would make better soap than the collected samples.

The values of the USM of both the reference sample and mean of the collected tallow sample are lower than that of the standard i.e. 12 g / kg in the tallow. Even though, in the case of tallow, as in the palm oil; the reference sample has lower USM value (1.33 g / kg) than the mean of the collected samples (4.7 g / kg).

The Iodine value (IV) is a measure of degree of unsaturation (C=C) in relation to the amount of fat or oil. Iodine value is defined as the gram of iodine absorbed per 100g sample. Hence, the higher the iodine value the greater the degree of unsaturation. The IV is used to characterize fat and oil, to follow the hydrogenation process in refining, and as an indication of lipid oxidation, since, there is a decline in unsaturation during oxidation (Pomerenz and Meloan, 1987). It is important as it gives the extent to which the lipid sample can be prone to oxidation and thus become rancid.

The iodine value obtained from the reference sample of the palm oil in this research (53.8) is within the range of the standard (45-55), indicating that it does not contain more than expected level of unsaturated fatty acid in the triglycerides. The mean of the collected samples (57.828) is a little above the maximum range of the IV in the standard. This implies that they contain more unsaturated fatty acids than the reference sample. A very high Iodine Value was obtained in sample C (66.4), and this could be attributed to the source and other ecological factors.

Fats are composed of more saturated fatty acids than oils, thus, it is expected that the IV of tallow to be lower than that of the palm oil. The IV of both the reference sample and the mean of the collected sample (44.15 and 42.785 respectively) are within the range of the standard (32-50). This indicates that both samples contain unsaturation within the standard specified, and that possibility of spoilage due to oxidation is within minimum range.

Saponins are phytochemical compound that comes as impurities in lipids and give rise to frothing in the sample. The determination of the Soap content (SC) parameter is to quantify the level of such contaminant. The SC in the reference sample is 0.003% m/m as compared with result in the mean of the collected samples which is exactly as the value for the standard (0.005% m/m). This indicates that both are free of saponins to minimal level of contamination.

The above is what goes for the results obtained in the tallow. Both the reference sample and the mean of collected samples' SC values (NIL and 0.004% m/m respectively) are lower than 0.005% m/m specified for the standard. Thus, are devoid of contaminant saponins.

As the case goes for SC, Insoluble Impurities (IM) are also contaminants in the fats and oil samples. For palm oil, results indicated that, both the reference sample and the mean of the collected samples' IM values (0.001 and 0.014% m/m respectively) are lower than the standard IM value i.e. 0.05% m/m. The same goes for results on tallow. As such, it can be concluded that insoluble matters are within accomodatable range in the samples analysed.

From the results obtained, approximately, same Titre value (TV) was obtained for reference sample and the mean of collected samples (≈41.5 ^{o}C). The values are slightly lower than that of the standard of 42 ^{o}C. In the case of tallow, results obtained showed that; both the reference sample and mean of collected samples TV (42.5 and 41.5 ^{o}C respectively) falls within the standard value range i.e. (40-49) ^{o}C. Although, the TV test have a limited use for edible fat or oil, but it is of great importance in soap and other industrial use where it play a significant role in bleaching.

Peroxide value (PV) measures the degree of lipid oxidation in fats and oils but not their stability. PV measures a transient (temporary) product of oxidation. A low value may represent early or advanced oxidation; which can be distinguished with time. For the determination of fats in foodstuff, it is difficult to obtain sufficient quantities from foods low in fat. The method is highly empirical; modifications may change results. Despite drawbacks, peroxide value is one of the most common tests of lipid oxidation (Nielsen, 2002).

PV is also used to check the presence of unsaturation just like the Iodine Value (IV). The PV in palm oil results obtained gave lower values than the standard (10 meq / kg) for both the reference sample and the mean of the collected samples (0 and 0.8 meq / kg respectively). In the same vein, tallow results are also lower than that of the standard i.e. 0 and 2.4 meq / kg respectively, as against 16 meq / kg in the standard.

Lipolysis is the hydrolysis of glyceride molecule into constituent fatty acids. Because of their volatility, production of short-chained fatty acids can result in off-odours. The term rancidity refers to as the offensive odours and flavours resulting from lipolysis (hydrolytic rancidity) or lipid oxidation (oxidative rancidity). It is also attributed to presence of or level of unsaturation in the lipid sample. Rancidity test is used to detect the extent of spoilage in fat and oil. (Nielsen, 2002).

Both the mean of collected samples and reference palm oils analysed are not rancid and confirms with the standard. Though, four of the collected samples (samples B, E, F and M) are rancid. In the case of tallow, the reference sample is not rancid, while the mean of the samples collected is against the standard, i.e. is rancid. But sample C is not rancid. The importance of this analysis is in many aspects of the food industry, ingredient technology, product development, quality assurance, product shelf life and regulatory aspects. The methods above help to characterize fat and oil and determine characteristics such as purity,

degree of unsaturation, average fatty acid chain length, adulteration level and status of the lipid with regards to oxidation.

Conclusion

The conclusion derived from the results of the analysis is that, some of the samples results do not comply with the standards; this may be due to adulteration or decomposition and the use of unripe fruits (in the case of palm oil) or combination of different animal fats.

Recommendation

It is recommended that regulatory bodies like National Food and Drug Administration and Control (NAFDAC), Standard Organization of Nigeria (SON) and other food quality regulatory bodies in the country should give routine check on fats and oils products within the country to ascertain their quality before they are sent to the market.

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