Physico-Chemical Analysis of Some Soaps Produced from Five Locally Processed Nigerian Oils

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Abstract

This study analysed the physical and chemical properties of five different types of soaps produced from five Nigerian locally processed Oils. In the physical analysis parameters such as the weight loss on curing, moisture content and foamability of the soaps were determined. In the chemical analysis, parameters such as saponification value and free fatty acid values of the soap were determined. The analysis uses standard methods of analysis employed widely. From the results it was discovered that within the variation of the experimental methods values of the parameters tested show close agreements with the values stated in the standard specification. Also, to a larger extent, the values are in close proximity with the results reported in previous literatures. It was therefore concluded that the oils used to produce the various soap samples conforms to the standard quality specification recommended for production of commercial soaps.

Keywords: Shea butter oil, palm kernel oil, palm oil, peanut oil, tallow oil.

1 INTRODUCTION

Soap is a cleaning or emulsifying agent made by reacting animal or vegetable fats or oils with potassium or sodium hydroxide. The reaction for making soap (saponification) is a base (usually NaOH or KOH) hydrolysis of triglycerides to make three salts (soap) and glycerol. The molecules crystallize differently depending on the base used. NaOH produces a harder bar while KOH is used more frequently for liquid soaps.

Oil + 3NaOH = Glycerol + 3 Soap [23]

Soap is a salt of a compound, known as a fatty acid. A soap molecule has a long hydrocarbon chain with a carboxylic acid group on one end, which has ionic bond with metal ion, usually sodium or potassium. The hydrocarbon end is non- polar which is highly soluble in non-polar substances and the ionic end is soluble in water. The cleaning action of soaps is because of their ability to emulsify or disperse water-insoluble materials and hold them in the suspension of water.

Soap often contains colouring matter and acts by emulsifying grease and lowering the surface tension of water so that it readily penetrates to remove dirt (Pius, et al., 2017). Normal soap is designed to decrease water's surface tension and to lift dirt and oils off the surfaces so that it can be easily rinsed away. It is a substance of ancient origin, the manufacture of which according to Gunstoneet et al. (1986) has evolved from primitive beginnings into a sophisticated chemical process.

The commonest oils used in soap making are lard and tallow from animal sources, coconut, palm oil, palm kernel, shea butter, groundnut oil, and olive oils from vegetable sources (Pavila et al., 1982).

There are several factors which affect the soap-making process and the soap quality. Such factors include the quality of oil, the amounts of the caustic soda and the amount of water used to make it. The reaction rate between oil and the caustic soda is influenced by free fatty acid content of the oil, the heat of the components before mixing, and how vigorously the mixing is to be done. Free fatty acid contents, vigorous mixing, and heat speed up the given soap-making process.

The physicochemical properties of soaps determine their quality and cleansing efficacy. Such physiochemical characteristics include pH, total fatty matter, free caustic alkali, moisture content, and free fatty acid among others. The qualities of alkali and oil used as well achieving complete saponification also have significant contribution to soap quality (Pius, et al., 2017).

1.1 TYPES OF OILS USED IN THIS STUDY

1.1.1 Shea Butter Oil

Shea Butter is from the nut of the shea tree, also known as "The Tree of Life". The Shea tree only grows in the savannah region of Africa. The Shea Tree produces its fruits once a year. The nut in the centre of the fruit when crushed and traditionally processed by boiling and extracting the oil, is what yields the vegetable fat known as Shea Butter (Poku, 2012).

The fatty acids present in the oil are oleic acid, linoleic acid, palmitic acid, stearic acid and arachidic acid (Boadu et al.,

2017). In Nigeria Shea butter tree is predominantly found in the northern part of Nigeria in states like Niger, kebbi, zamfara, and Kwara states. The oil of shea butter tree is mostly processed locally and is used as fuels for lightening, skin ointment and for other medicinal purposes.

1.1.2 Palm Kernel Oil

Palm kernel oil is edible plant oil derived from the kernel of the oil palm (Poku, 2002). Palm kernel oil is one of the few highly saturated vegetable fats, which give the name to the 16-carbon saturated fatty acid, palmitic acid (8-9%) that it contains ($C_{15}H_{31}COOH$). Other fatty acids contained in palm kernel oil are lauric (47-48%), oleic (15-18%), myristic (14-16%), capric (4%), stearic (3%) and linoleic (2%) acids. This oil is semi-solid at room temperature, is more saturated than palm oil. It is commonly used in commercial cooking because of its relatively low cost, and because it remains stable at high cooking temperatures and can be stored longer than other vegetable oils [20].

1.1.3 Palm Oil

Palm oil is produced from the fruit pulp of the oil palm tree (*Elaeisguineensis*). This tree is native to the area of West Africa near the Gulf of Guinea, which is where its scientific name comes from. These tropical fruits are rich in oil (45-65%), and are naturally reddish in colour because of the fruit pulps' high carotenoid content. The fruit has a single seed or kernel, which is used to produce different oil, called palm kernel oil, which has a completely different structure from palm oil (fruit palm oil). Palm oil is extracted by heating and pressing the pulp of the fruit. The refining process is applied to purify the crude palm oil, providing highly versatile oil, with important functional properties. The fatty acids contained in palm oil include palmitic (43-45%), Oleic (38-40%), linoleic (9-11%), stearic (4-5%) and myristic (1%) acids.

1.1.4 Peanut/Groundnut Oil

Groundnut oil is derived from well groundnuts (Arachishypogaea) and is important source of energy in our diet, meeting specific nutritional requirements. It works well with a wide variety of foods, which is a good thing because it contains heart friendly MUGFA (monounsaturated fatty acid). Groundnut oil is also low in saturated fats. Its high smoking point means the oil holds to its nutritional content at high temperatures. Some of the fatty acids present in the oil are oleic (48%), linoleic (32%), palmitic (11%) and stearic (2%) acids.

Groundnut oil or peanut oil is abundantly grown in the northern parts of Nigeria.

1.1.5 Tallow (Beef) Oil

Tallow is an animal fat obtained by rendering animal carcases (sheep or beef) and waste from the food industry. Like vegetable oils, tallow is a triglyceride. A triglyceride consists of a three carbon glycerol head group to which are added three fatty acid chains. All triglycerides have the same basic structure, and the differences in properties and use of commercial triglycerides depend entirely on the length, degree of unsaturation and other chemical modifications to the fatty acid chains (Piaszyk et al., 2010). Some of the fatty acids obtainable in tallow oil are oleic (37-43%), Palmitic (24-32%), Stearic (19-25%), myristic (3-6%) and linoleic (2-3%).

1.2 Aim and Objectives of the Study

The major objective of this study is to establish the qualities of some soaps produced from five Nigerian locally processed fatty acid oils using the physic-chemical properties of the soaps and oils. The physico-chemical parameters used are: saponification value, free fatty acid test, moisture content test, foamability tests and weight loss test (curing process).

2.0 MATERIALS AND METHODS

2.1 Materials

2.1.1 Samples Collection

The tallow (animal fat) sample was collected directly from the abattoir. The plants fats (shea butter, palm kernel, palm oil and peanut oil) were obtained in their pure form from the market.

2.1.2 Samples Preparation

All the samples except the tallow fat were collected in liquid form. The tallow oil was collected in solidified form and was melted before saponification with the lye solution.

2.1.3 Soaps production

The soap samples were produced by mixing a standard prepared quantity of lye with a known quantity of the oil. The standard lye solution was prepared by calculating the quantity of caustic soda that would saponify each sample of the oil.

Table 1 below are the standard saponification values of the oils used to calculate the quantity of caustic soda needed to saponify given quantity of each oil sample..

Table 1: Standard Saponification Values of the Oils.

OIL SAMPLE	SAPONIFICATION VALUE(Mg(NaOH)/g)
SBO	0.128
PKO	0.156
PMO	0.141
PNO	0.136
TLO	0.138

Source: Michelle, 2009

2.1.4 Calculations of Amount of Caustic Soda (NaOH) Used in Soaps Productions

The quantity of caustic soda used in the production of the soaps was determined using the mathematical relation below:

$$\text{Qty (NaOH)} = \frac{200 \text{ x StdS.V(Oil)}}{1} \qquad (1)$$

Where,
Qty (NaOH) = quantity of NaOH used
200 = the quantity of oil used
StdS.V(Oil) = the standard saponification value of oil.
1 = number of gram of oil saponified by 1g of NaOH

From equation (1) the quantity of caustic soda used to saponify 200g of different oil is calculated and presented in table 2 below:

Table 2: Quantities of Caustic Soda and Water used to produce the Oil Soaps.

OIL SAMPLES	QUANTITY OF NaOH (g) USED	QUANTITY OF WATER USED TO PRODUCE 25% NaOH SOLUTION (CM^3)
SBO	25.6	76.8
PKO	31.2	93.6
PMO	28.2	84.6
PNO	27.2	81.6
TLO	27.6	82.8

SBO=Shea butter; PKO=Palm kernel; PMO=Palm Oil; PNO=Peanut Oil; TLO=Tallow Oil

2.1.5 The Water/NaOH Ratio

Water does appear in the saponification reaction. There is no fixed relationship between the numbers of molecules of water needed for a molecule of oil [23]. Water is basically used to dissolve the sodium hydroxide so that it can react with the oil.

However, there is the need to determine the standard value for the amount of water to be used in the saponification of the oils.

Many previous studies have reported on the percentage value of the concentration of sodium hydroxide (NaOH) to be used in the saponification reaction. Cavith, (1997) recommends a concentration of 30% as the starting value with an average concentration of 27%. Both Branson, (1972) and Cavith adopted concentration of 26-27% and is the reason why sodium hydroxide calculators use 27% as the normal, correct, standard lye concentration 23].

The amount of water used in soap making affects the rate at which the saponification reaction and the curing process take place. When the amount of water is less the rate of saponification and the curing process is faster and vice versa (Watson, 2007).

Also, when the concentration of the mixture is high (i.e. less water) it becomes harder to dissolve the sodium hydroxide and the solution may give off more fumes [23].

In this study we decided to adopt a concentration of 25% as the alkali-water mixture as a starting point. This gives us a ratio of 1:3 between the amount of caustic soda and the amount of distilled water to be used.

Using the quantity of caustic soda values in table 2 the corresponding quantity of distilled water needed to make up 25% of the lye solution was obtained by multiplying with a factor of 3. These quantities for oil samples are presented in table 2.

2.1.6 Processing of the Soaps

The cold method of production was used for the production of the soaps. 200g of oil was slowly poured into the corresponding 25% sodium hydroxide (lye) solution and stirred repeatedly to form liquid paste. The paste was stirred continuously until the thickness of the paste increases and a trace-mark begins to appear. The paste in then transferred into a plastic silicon mould and allowed to cure at ambient temperature into a solid rectangular soap bars.

Table 3: Compositions of the Oil Soaps

SOAP SAMPLE	COMPOSITION
SBO	Oil200NaOH25.6H_2O76.8
PKO	Oil200NaOH31.2H_2O93.6
PMO	Oil200NaOH28.2H_2O84.6
PNO	Oil200NaOH27.2H_2O81.6
TLO	Oil200NaOH27.6H_2O82.6

2.2 METHODS

2.2.1 Physical Analysis

2.2.1.1 Determination of Foamability

About 2.0g of each soap (shavings) was added to a 500cm^3 measuring cylinder containing 100 cm^3 of distilled water. The mixture was shaken vigorously so as to generate foams. After shaking for about 2 minutes, the cylinder was allowed to stand for about 10 minutes. The height of the foam in the solution was measured and recorded (Isah, 2006).

2.2.1.2 Determination of Moisture Content

Moisture content was determined by drying 10g of the sample to a constant weight at 105 ºC according to AOAC (2000). It was allowed to cool and then reweighed. The moisture content was determined by from the following formula:

Moisture content = $(C_s - C_h / C_s - C_w) \times 100$
Where;
C_w= weight of crucible
C_s=weight of crucible + sample
C_h = weight of crucible + sample after heating (Ashrafy et al. 2016).

2.2.1.3 Determination of Weight Loss on Curing

The samples were allowed to cure at room temperature for 1 week. During that time the weight of the samples were taken after every 24 hours interval. The weight loss was determined using the following formula:

Weight loss = $W - W_1$
where;
W = Initial weight of the sample
W_1 = weight of the sample each day.

2.2.2 Chemical Analysis

2.2.2.1 Determination of the Saponification Value (SV)

The saponification value of the oils were determined by weighing 2g of the oils and placed into a 300 cm^3 conical flask. 0.5M solution of KOH was added to the solution and heated to 55°C over water bath with continuous stirring. The temperature was raised to 100°C to complete the saponification process. The mixture was allowed to boil for about 1hour. The excess KOH was titrated against the mixture using phenolphthalein indicator until pink color was observed (Aiwizea et al., 2012).

The Saponification Value (SV) was determined using the following formula:

$$S.V = \frac{56 \times N(KOH)(V_2 - V_1)}{W}$$

where;
SV= saponification value
W = weight of the oil sample
N = actual normality of KOH
V_1 = volume of HCl used in real titration
V_2 = volume of HCl used in the blank

ii) **Determination of Free Fatty Acid (FFA)**

Free fatty acid is defined as molecules that are long chains of lipid of carboxylic acid found in fats and oils.

1g of oil was boiled with 50ml of ethanol and allowed to cool. 2 drops of phenolphthalein indicator was added to the mixture. The mixture was then titrated against 0.1N NaOH until pink color was obtained (AOAC, 1997). The free fatty acid was calculated from the formula below.

$$\%FFA = \frac{Tx22}{W}$$

Where;
FFA = Free fatty acid
W = Weight of sample
2.82 = Molecular mass of sodium Hydroxide
T = Average titre value

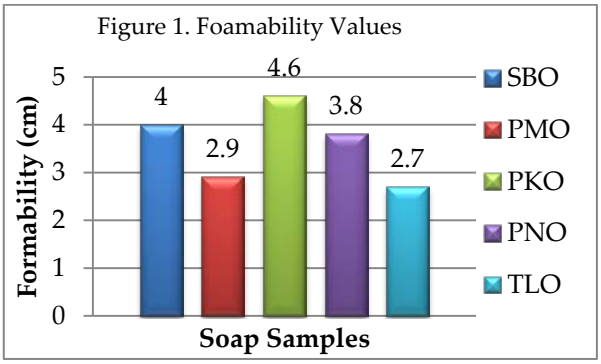
3.0 RESULTS AND DISCUSSION

3.1 Physical Analysis

3.1.1 Foamability Tests

Figure 1 below is the bar chart plots of the foamability tests carried out on the soap samples.

Figure 1. Foamability Values



The bars represent the heights of the foams formed after the soaps were dissolved in distilled water and shaken vigorously for 2 minutes.

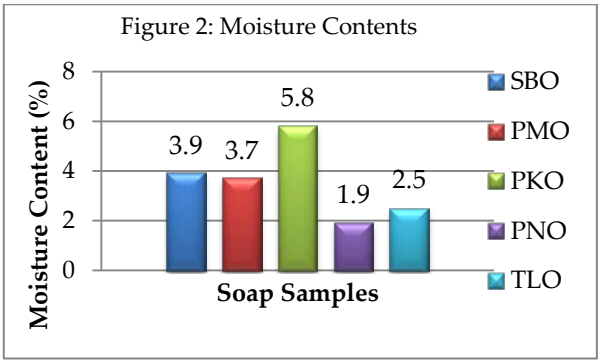
The bars in figure 1 show that the amount of increases in the foamability of the samples increases in the order PKO > SBO > PNO > PMO > TLO. It means that palm kernel produces the highest amount of foam and tallow oil produces the lowest amount of foam amongst the samples.

The foamability values of SBO and PNO are above the formability values of shea butter oil and peanut oil reported elsewhere [7]. But, that of TLO soap falls within the same range of 2.7 max as reported [7].

3.1.2 Moisture content

The values of the moisture content of the samples are presented in figure 2 below. The moisture content was determined two weeks after the soap was produced. The chart shows that the increase in the moisture content of the samples are in the order PKO > SBO > PMO > TLO > PNO.

Figure 2: Moisture Contents



For SBO sample the moisture content value is close to the value of 3.7% reported by Zauro et al., (2016), in which the test was conducted eight weeks after the soap samples were produced. However, the moisture content value obtained for SBO exceeds in this study is lower than the value of 9% obtained by a similar investigation [15]. This variation may be as a result of time difference in which the tests were carried out.

In the case of PKO, the moisture content (5.8%) obtained in study is lower than what was reported by other researchers in other literatures [14](14%), [17](41%). Aldo as reported in a similar investigation the moisture content of certain commercially produced soaps was determined to be between 10.91%- 22.69% [18].

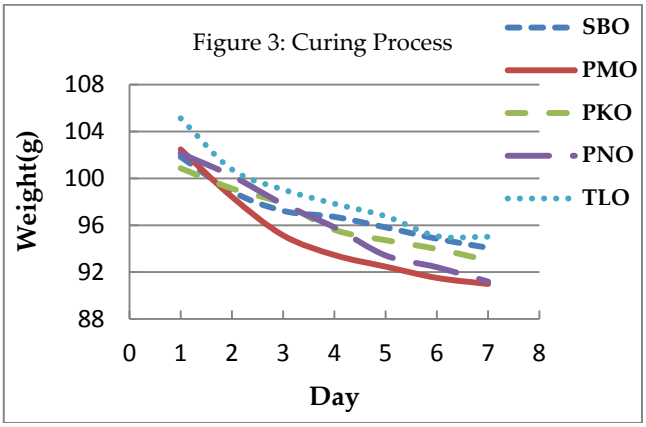
Variation in moisture content could be associated to the variation in the methods of soap production employed by different author.

Variation in the moisture content values reported in various literatures could be associated with the differences in the production methods adopted by each scholar. In addition, it could also be attributed to the time frame in which the test was carried out. Time frame plays a crucial role in the determination of moisture content of soaps. This is because as the soap undergoes curing process water in the soap is continuously being lost through evaporation [23].

As such, any delay in conducting the moisture test would affect the quantity of water content of the soap. As such for accurate comparison of the moisture contents there is the need to consider the time variation in which the tests were conducted, otherwise the comparison will be implausible.

3.1.3 Hardening (Curing) Process

Figure 4 is the curing process of the oil soap samples. The curves for all the samples exhibit an exponential decay in the weights of the samples during the curing process. This means as the curing time progresses the weight of the soaps samples decreases gradually until it reaches a constant weight and the soap is said to dry and all the water molecules are eliminated. Eventually the weights approach constant values at time progress.



After 1 day the PKO soap has the lowest weight of all the soap samples with a recorded weight of 100.8g. It will take SBO soap 1.32 days, PMO soap 1.36 days, PNO soap 1.62 days and TLO 2 days to achieve this weight.

From table 2 the volume of water used to form 25% of the lye solution for the production of PKO soap sample is the largest compared with the other soaps samples. However, in terms of weight loss, PKO soap loses the largest amount of water after 1 day compared with the other soaps. As such, the rate of loss of water within the first one and a half days of the soap is larger for PKO soaps than the other samples. However, after that the rate of loss of water of PMO exceeds that of the other soap samples. This continues up to the 7 days mark. The TLO soap produces the least rate of water loss of all the samples from day 1 up to day 7.

Table 4: Rate of Weight and Water Loss of the Samples during Curing

Sample	Weight loss on Curing (g)/7 days	Water loss (%)
SBO	7.75	42
PKO	7.87	50
PMO	11.49	68
PNO	10.95	67
TLO	10.11	61

Table 4 represents the weight loss and percentage water loss of the samples. The increase in weight loss of the samples is in the order PMO > PNO > TLO > PKO > SBO. The decrease in percentage of water loss of the samples is in the order SBO > PKO > TLO > PNO > PMO. As such PMO has the highest rate of

water loss upon curing compared with the other samples.

Table 5: Theoretical, Standards and Experimental Saponification Values of the Samples

SAMPLES	THIS STUDY SAP. VALUE MgKOH/g	FAO STANDARD SAP. VALUE (MgKOH/g (FAO STANDARD)	STANDARD SAP. TABLE MgKOH/g (MICHELLE, 2009)	THEORETICAL SAP. VALUE (MgKOH/g	STANDARD DEVIATION
SBO	220.13±1.36	199-240	179.2	194	17
PKO	241.18±1.40	199-240	218.4	218	10
PMO	169.84±0.03	185	197.4	200	12
PNO	184±0.06	189	190.1	193.6	3
TLO	133.09±0.10	171	196.7	200	27

SAP = saponification

3.2 Chemical Analysis

3.2.1 Saponification Value

The results of the saponification values of the five samples are presented in table 5 above.

The table shows the experimental saponification values of the samples, the saponification values according to the Food and Agricultural Organisation (FAO) standard specification, saponification values according to standard saponification table (Michelle, 2009) and theoretically computed saponification values.

The theoretical saponification values of the oil samples were computed using the equation reported by Kevin, (2008), below:

$$? \text{ g KOH} = 1000g\left(\frac{1molOil}{\frac{X\ g}{Oil}}\right)\left(\frac{3molKOH}{1molOil}\right)\left(\frac{56\ \frac{g}{KOH}}{1molKOH}\right)$$

where, X is the molar mass of the triglyceride.

Statistical analysis of the data in table 5 shows that the standard deviation of the individual samples across the entire spectrum of the data is large; with the exception of PNO samples

In addition, the saponification value of PMO falls short of the values of 191.32 and 196.3-207.22 previously reported [8], [10].

In the case of the PKO sample although the saponification value reported in this study is within the range stipulated by FAO, it is, however, below the value of 280.5 reported by Amira et al. (2014).

The saponification value of TLO compares very well with the reported value of 140.3 reported by Warra et al., (2010), but it is lower than the value of 190.73 as reported by Abdulkadir et al. (2013).

The saponification value of PNO compares well with the values reported by Warra et al. (2010) (187.7). It is, however, significantly lower than the values reported by Nkafamiya et al. (2010) (220.20) and Amira et al. (2014) (257.5)

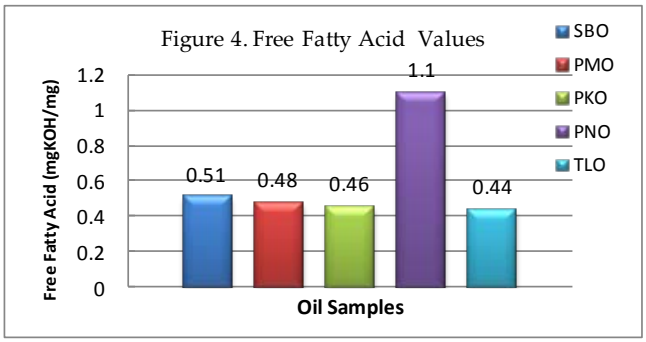
In the case of SBO the saponification value is within the value reported by Chibor et al. (2017) (227.94), but it is considerably higher than the values reported in other studies conducted by Ogbonnaya et al. (2008) (196.9); Okullo et al. (2010) (160.0) and Warra et al. (2010) (183.9).

It has been reported that the recommended value for saponification value is between 195-205 [27] and it a measure of the suitability of oil for industrial use [25]. The higher the saponification value the greater the effectiveness of the oil for industrial production.

From the saponification value obtained in this study the increase in the order of saponification value of the oil is in the order TLO < TMO < PNO < SBO < PKO. This means that the suitability of the oils for industrial use shows that Palm kernel oil is the most suitable. The trend in the order of suitability follows the foregoing reverse order.

3.2.2 Free Fatty Acid

Figure 5 is the bar chart plots of the free fatty acid values of the oil soap samples. There are, however, variation differences between the values obtained under this study and the values reported by previous authors (see table 5)



The plots show that the free fatty acid values of the all the oils compare well with the FAO standard values (SBO < 0.5; PKO < 0.5; PMO < 0.5; PNO < 1-2; TLO < 0.5).

Table 6: Free Fatty Acids from Various Investigations

SAMPLES	FREE FATTY ACID VALUE (mg(KOH)/g									
	This Study	Chibor et al. (2017)	Abdulkadir et al. (2013)	Akinola et al. (2010)	Nkafamiya et al. (2010)	Musa et al. (2012)	Hee Seung Nahm (2011)	Amira et al. (2014)	FAO Standard	SON
SBO	0.51	0.85	2.12						< 0.5	
PKO	0.46							1.35	< 0.5	
PMO	0.48			0.28-1.31					< 0.5	3.5
PNO	1.10				1.55-3.95	1.32-3.00	1.07-8.56		1.2	
TLO	0.44								< 0.5	

Free fatty acid is the percentage by weight of a specified fatty acid (e.g. percent oleic acid) [20]. High concentrations of free fatty acids are undesirable because FFAs are odorous substances producing irritation on the tongue and in the throat. They make oil unsuitable for consumption depending on their concentration. If they occur in minute quantity, their effect may become unnoticeable [16].

The recommended free fatty acid value according to FAO standard is as indicated in table 5 above. The quantity of free fatty acid in oil is an indicator of its overall quality. High value of free fatty acid in oils indicates poor quality of the oil and could result from contamination with impurities that could cause the hydrolysis of the ester linkage thereby increasing the free fatty acid level [20]. Low level of free fatty acid indicates good quality of the oil and renders the oil from becoming rancid and odorous. It also indicates the suitability of the oil for use in the industry.

From the values of the free fatty analysis carried out on the samples used indications show that there are good fittings between the values for the samples and the standard values recommended by FAO (table 5).

The results show that the suitability of the oil for consumption and industrial applications is in the order: PNO > SBO > PMO > PKO > TLO.

4 CONCLUSION

From the results obtained both of the physical and chemical parameters it can be concluded that the physical and chemical properties of the oils and soaps tested are within the range of the standard values recommended by standard specification (FAO standard). Although there are variation with the values of the parameter with other reported investigation, however, these variations are not peculiar to this results obtained in this study. Observation has shown that the variations are widespread even amongst the results of the investigations conducted previously. To this end, we can assert conclusively, within a reasonable degree of experimental errors, that the locally processed oils used in this investigation can produce good and quality soaps that conform to any standard.

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