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CHEMICAL ANALYSIS AND BASE- PROMOTED HYDROLYSIS OF LOCALLY EXTRACTED SHEA NUT FAT

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ABSTRACT
The study was on the chemical analysis and base- promoted hydrolysis of extracted shea nut fat. The local method of extraction of the shea nut oil was employed in comparison with literature report. A simple cold-process alkali hydrolysis of the shea nut oil was used in producing the soap. The chemical analysis of the oil revealed that it had saponification, iodine and acid values of* 136.32 ±1.943 mgKOH/g, 50.50 ± 8.023g I_2/100g *and* 14.77 ± 0.065 mgKOH/g *respectively. The analytical values obtained were significantly in favour of the utilization of the indigenous shea nut fat for soap production on commercial scale. The pH of the soap was 10.33, which is in agreement with the pH range of 9-11 set by the National Agency for Food and Drug Administration and Control(NAFDAC), due to incomplete alkali hydrolysis resulting from the saponification process. The foam height of the soap was 4.2cm lower than that of Jatropha, sesame and cotton seed soaps analyzed higher than that of Neem, castor and castor superfatted with glycerine soaps. The soap was white and slightly soluble in distilled water.
***Key words*:** Shea nut fat, chemical analysis, Saponification, Foam ability and Solubility.

INTRODUCTION

The shea nut fat is produced from shea nuts derived from the shea nut tree, formerly *Butryospermum paradoxum*, now called *Vitellaria paradoxa*.

This tree is an indigenous tree species to many countries in Sub-Saharan Africa and in these countries the shea tree and its many uses have been known for over centuries.(Carette *et al.*, 2009). It constitutes an important source of fat in food and cosmetics.(Okullo *et al.*, 2010). Its fatty matter has been used for years in Africa for different purposes, ranging from food and soap processing, to healthcare and other medicinal uses.(Coulibaly *et al.*,2009). It is also used to treat horses internally and externally for girth galls and other sores. Sheanut oil is composed of triglycerides (triacylglycerols) containing an oleic acid moiety at the 2-position and saturated fatty acids, usually stearic or palmitic acids, at the 1- and 3-positions. Shea butter is highly regarded in the cosmetic field because of its high emmoliency and moisturizing capacities (Acquaye *et al.,* 2001).

Chemical composition

Chemical analysis of Shea butter extracted from nuts samples from four African countries (Uganda, Nigeria, Burkina Faso and Mali) were conducted by the Ben Gurion University, Isreal, as part of the ongoing EU funded INCO project on Shea. Fatty acid analysis shows there is a high level of variability in Shea oils across Africa. The Ugandan sample had a 59% oleic acid content compared with 47% for Nigeria and only 39% for Burkina Faso.(Ferris *et al.,*2001).

This work is aimed at preparation of soap from locally extracted Nigerian shea nut fat and analyze its physical and chemical characteristics.

MATERIALS AND METHODS

Shea nut fat extraction: The shea nut was obtained from Kwanga town in Ngaski Local Government Area of Kebbi State and was extracted using local extraction method (Warra, 2009; Khobe *et al.,* 2009). This method is cheaper and better method to extract shea butter because there is no need for chemicals or synthetic agents added. This process has been in practice for many years by the natives of Gungawa tribe and as reported by (Warra, 2009; Khobe *et al.,*2009) requires the following steps;

(1)The nuts were Par-boiled to prepare them for shelling followed by drying and removal of the bad ones.
(2) The nuts were crushed by grinding into a powdery material and was further milled using milling machine
(3) The milled flour was mixed with cold and hot water to break the emulsion and to facilitate separation.
(4) Separation of the fat was done using cold-water separation process causing the fat to float.
(5) The shea fat was decanted leaving a brown residue devoid of oil, which settled to the bottom.
(6) The butter was dehydrated by boiling leaving it to settle under the pot**.**
(7) The shea fat was purified by decanting the liquid fat, which was then allowed to cool and solidify.

These steps can be compared relatively with the method reported (Khobe *et al.,* 2009)

To avoid rancidity the shea butter sample was stored and transported in airtight plastic containers.

Shea oil analyses: The chemical analysis of the oils was carried out using the methods adopted by AOAC (1998), Akpan *et al.* (2006) and Bassir, (1978).

Saponification value: About 2 g of the oil sample was added to a flask with 30 cm^3 of ethanolic KOH and was then attached to a condenser for 30 minutes to ensure the sample was fully dissolved. After sample had cooled, 1cm^3 of phenolphthalein was added and was titrated with 0.5M HCl until a pink endpoint has reached.

Saponification value was calculated from the equation

$$SV = \frac{(S-B) \times M \times 56.1}{\text{Sample weight (g)}}$$

Where S = sample titre value
B = blank titre value
M = molarity of the HCl
56.1 = molecular weight of KOH

Iodine value: The iodine value was determined according to the standard method of Akpan *et al.,* 2006. 0.4 g of the sample was weighed into a conical flask and 20 cm^3 of carbon tetra chloride was added to dissolve the oil. Then 25 cm^3 of Dam's reagent was added to the flask using a safety pipette in fume chamber. Stopper was then inserted and the content of the flask was vigorously swirled. The flask was then placed in the dark for 2 hours 30 minutes. At the end of this period, 20cm^3 of 10% aqueous potassium iodide and 125cm^3 of water were added using a measuring cylinder. The content was titrated with 0.1M sodium-thiosulphate solutions until the yellow colour almost disappeared.
Few drops of 1% starch indicator was added and the titration continued by adding thiosulphate drop wise until blue coloration disappeared after vigorous shaking. The same procedure was used for blank test and other samples.
The iodine value (I.V.) is given by the expression

$$I.V. = \frac{12.69C\,(V_1-V_2)}{M}$$

Where C = Concentration of sodium
V_1 = Volume of sodium thiosulphate used for blank
V_2 = Volume of sodium thiosulphate used for determination
M = Mass of the sample.

Acid value: 100 ml of neutral ethyl alcohol was heated with 10 g of oil or fat sample in a 250 cm^3 beaker until the mixture began to boil. The heat was removed and was titrated with N/10 KOH solution, using two drops of phenolphthalein as indicator with consistent shaking for which a permanent pink colour was obtained at the end point.
The Acid value was calculated using the expression; A.V = 0.56 x No. of ml. N/10 KOH used.

Soap preparation: The alkali solution used was prepared by dissolving 200 g of sodium hydroxide pellets in 1 dm^3 volumetric flask and the volume made to the mark with water. For the soap formulation, the method reported by Warra (2009) was used. 200 g/dm^3 alkali solution was poured directly into 200 cm^3 beaker containing shea nut oil in the ratio 1:1 (v/v) of the mixture. The solid shea nut fat was warmed gently in a beaker using hot plate so that it will be in liquid state (at room temperature) and was poured into the 200cm^3 beaker followed by the alkali solution and then stirred frequently for 10-15 minutes using a stirring rod. The thickened mixture was then poured into a wooden mould and allowed to harden by air-drying for 24 hours to obtain the soap bars.

pH Determination:
The pH of the various soaps produced was determined using a pH meter (827 pH lab Model). 10g of the soap shavings was weighed and dissolved in water in a 100cm^3 volumetric flask. This was made up to prepare 10% soap solution in line with literature report (Dalen and Mamza, 2009). The pH reading was recorded. The steps were repeated using various soap samples produced.

Foam ability Tests
Although foam generation has little to do with cleansing ability (Mainkar and Jolly, 2000), it is of interesting importance to the consumer and is therefore considered a parameter in evaluating soaps and detergents.
Mainkar and Jolly (2000) mentioned commonly used test protocols for foam test. The pour foam test developed by Ross and Miles (1941). Hart and Degeorge (1980) preferred to measure the lather drain times, whereas Sorkin *et al.*, (1966) called for rotating a shampoo solution in a glass stoppered cylinder. Neu (1960) used kitchen blender to produce foam and reported that the foam characteristics were similar to those observed in practice. We used the method reported by Isah, (2006) for synthetic detergent. About 2.0g each of soap (shavings) was added to a 500cm^3 measuring cylinder containing 100cm^3 of water. The mixture was shaken 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 of the various soap samples used was measured and recorded.

RESULTS AND DISCUSSION

Table 2: Physicochemical characteristics of the indigenous shea nut fat

Parameter	Observation
Saponification value mgKOH/g	136.32 ±1.943
Iodine value g I_2/100g	50.50 ± 8.023
Acid value mgKOH/g	14.77 ± 0.065
Physical state at room temperature	Solid

The values are mean and standard deviation of triplicates determinations.

Table 3: Results of the physical and chemical characteristics of the prepared shea butter soap.

Parameter	Observation
pH	10.33
Foam height (cm)	4.2
Colour of soap solution	White
Solubility in water	Slightly soluble

The values are mean of triplicate determinations.

Table 4: pH of the various soap samples of the present research

Soap sample	pH value
Castor oil based soap	9.70
Castor glycerine soap	9.60
Cotton oil soap	9.38
Jatropha oilbased soap	10.11
Neem oil	9.90
Sesame oil soap	9.88
She nut fat soap	10.33

The values are mean of triplicate determinations.

Table 5: Foam ability as a function of foam height of the various soap samples of the present research

Soap sample	Foam height (cm)
Castor oil based soap	1.6
Castor glycerine soap	1.4
Cotton oil soap	4.5
Jatropha oilbased soap	5.4
Neem oil	2.0
Sesame oil soap	4.8
Shea nut fat soap	4.2

The values are mean of triplicate determinations.

DISCUSSIONS

The physicochemical analyses carried out on indigenous crude shea fat (Table 3), was relatively similar to the literature values (Mabrouk, 2005; Oyedele, 2002) determined for the indigenous crude shea fat. Saponification value of 136.32 ±1.943 mgKOH/g obtained was lower than that of olive oil (192 mgKOH/g) and sunflower oil (188.7 mgKOH/g) but higher than that of beeswax (93 mgKOH/g), which are used in soap making (Mabrouk, 2005). This indicates that the oil could be used in soap making since its saponification value falls within the range of these oils. Higher saponification justify the usage of fat or oil for soap production.

Iodine value of 50.50 ± 8.023g I_2/100g (less than 100) was obtained, which shows that the oil belongs to the class of Non-drying oils, which are useful in the manufacture of soaps (Kochhar, 1998).An Acid value of 14.77 ± 0.065mgKOH/g was obtained which is lower than that of olive oil 17 mgKOH/g (Davine and Williams,1961) higher than 10.49 3mgKOH/g reported by Oyedele for shea nut fat (2002), which made it suitable for soap production.

For the prepared soap the pH was 10.33 (Table 4) comparably within the higher pH range of 9-11 but favourably higher than the pH range of 3-5, which are considered as high and low levels respectively by the National Agency for Food and Drug Administration and Control(NAFDAC), (Umar,2002) mostly due to incomplete alkali hydrolysis resulting from the saponification process. This can be overcome by the addition of excess fat or oil or any other superfatting agent to reduce the harshness of the soap. Superfatting soaps with 1-2% neutral oils or glycerine also resulted in the better quality of soaps that were free of cracks(Kuntom *et al.*, 1999).The foam height of the soap was 4.2cm(Table 5) lower than that of Jatropha, sesame and cotton seed soaps, higher than that of Neem, castor soap and castor superfatted with glycerine soap. The soap was white and slightly soluble in distilled water.

CONCLUSION

From the results obtained after the chemical analysis of the fats it can be concluded that the selected fat is utilizable for soap making. The properties exhibited by the soap solution indicated its suitability for commercial production.

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