



Fat Quality and Cold Saponification of Shea Nut (*Vitellaria paradoxa*) Fat Extract

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Authors' contributions

Author AAW designed the study, wrote the protocol, and wrote the first draft of the manuscript. Author JIK managed the analyses of the study and some of the literature searches. Interpretation and write up of the manuscript were done by authors AAW and JIK. All authors read and approved the final manuscript

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ABSTRACT

Aims: To extract, carry out chemical analyses and saponify shea nut fat.
Study Design: Triplicate Chemical analysis using standard procedures was employed.
Place and Duration of Study: Biochemistry Laboratory, Department of Biochemistry, Kebbi State University of Science & Technology, Aliero, Nigeria.
Methodology: Shea nut fat was extracted in a soxhlet apparatus using analytical grade hexane (n- hexane) as refluxing or extracting solvent for the work. At the completion of the extraction process the oil was recovered from the mixture by evaporating the residual extracting solvent in an oven set at 50°C and stored in the bottle. This process was repeated until a substantial quantity of oil was achieved. Each batch of extraction lasted for about 5 hours on the average. Standard reported procedures were used to carried out the chemical analyses.
Results: The powdered seed gave 38.74% oil with density of 0.96g/ cm³. The oil is yellowish in color before it solidified to fat at room temperature. Chemical analysis of the oil reveals saponification value 166.10±0.84mgKOH/g, iodine value53.54±0.39g I₂/100g, acid value of 14.26±0.33mgKOH/g and free fatty acid value of 4.20±0.04. A simple cold-process alkali hydrolysis of the shea nut oil was used to produce a brown colored soap with a foam height of 31cm³. The chemical properties of the soap were 58.66 % total fatty matter , 0.57 % total alkali, 1.26 % percentage chloride, and pH of 10.7.

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Conclusion: Due to the favorable chemical characteristics of the soap in comparison with literature reports, it can be used as cosmetic toilet soap especially when other ingredients such as sequesterants, silicates and glycerine are added.

Keywords: Sheanut; extraction; fat extract; saponification; quality.

1. INTRODUCTION

Shea nut fat is a byproduct of nuts obtained from shea tree also known as *Vitellaria paradoxa* [1] or *Butyrospermum parkii* which belongs to the *Sapotaceae* family. It constitutes an important source of fat in food and cosmetics [2]. 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 [3]. Global interest in the product stems from its use as a cocoa butter equivalent in the pharmaceutical and cosmetics industries [4]. It is also used as an unguent for the skin and has anti-microbial and soothing properties, which give it a place in herbal medicine. It is also used as an important raw material and/or a precursor for the manufacture candles [5]. The fat obtained from the shea kernel is referred to as shea butter and it is the most valued product from the shea tree [6]. The aim of this research was to extract, carry out chemical analyses and saponify shea nut fat.

2. MATERIALS AND METHODS

2.1 Sample Preparation

The *Cyperus esculentus* L .tubers which was procured from local tiger nut traders at Tawa market, Niger Republic were air-dried in the laboratory at room temperature, for a period of one week, and was then ground, using pestle and mortar. The dry powdered sample was then kept at room temperature in the laboratory for extraction.

A matured shea nut fruit was collected from Kwanga town of Ngaski local government, Kebbi State, Nigeria, the fruits were collected with leaves of the shea nut tree for taxonomic identification. It was authenticated by Dr. Dhramendra Singh of the Botany unit Biological Sciences Department, Kebbi State University of Science and technology, Aliero in comparison with voucher specimen No. 320 kept at Herbarium. The nuts were scooped out of the fruit and washed with distilled water and were allowed to dry. The kernels were removed and dried under the sun for two days and were ground with agar mortar and pestle. The powdered sample was then kept at room temperature in the laboratory for extraction.

2.2 Oil Extraction

The extraction of oil from the tubers was carried out in a soxhlet apparatus using analytical grade hexane (n- hexane) as refluxing or extracting solvent for the work. At the completion of the extraction process the oil was recovered from the mixture by evaporating the residual extracting solvent in an oven set at 50°C and stored in the bottle. This process was repeated until a substantial quantity of oil was achieved. Each batch of extraction lasted for about 5 hours on the average [7].

2.3 Determination of Colour

The colour of the oil samples was determined by observation using several independent competent individuals. Oil colour was correlated using colour charts [7].

2.4 Determination of Percentage Yield

The hexane extract of the shea nut fat was transferred into a measuring cylinder which was placed over water bath for 30 min at 70°C so as to ensure complete evaporation of solvent. Then percentage yield was calculated as follow.

$$\text{Oil content (\%)} = \frac{\text{Weight of the oil}}{\text{Weight of sample}} \times 100$$

2.5 Determination of Specific Density

This was performed according to literature report [8]. The 10ml of the oil was measured in a pre-weighed measuring cylinder. The weight of the cylinder and oil was measured; the weight of the oil was then obtained by subtracting the weight of the cylinder from the weight of the oil and cylinder. The specific density of the oil was obtained using equation below.

$$\text{Density of oil} = \frac{W_1 - W_o}{V_o}$$

Where W1 = weight of empty measuring cylinder + oil. W_o = weight of measuring cylinder, V_o = volume of oil used.

2.6 Determination of Saponification Value, Acid Value, Iodine Value and Free Fatty Acid

These were performed according to standard methods of analysis [9].

2.7 Analysis of Soap Produced

2.7.1 Saponification procedure

As reported in literature [10], for each soap formulation, alkali solution were poured directly into the beaker containing the oil in the ratio 1:1 (v/v). The oil was warmed gently and poured into the beaker followed by the alkali solution to form an intimate mix and then stirred frequently for some minutes using stirring rod. The saponification mixture was then poured into moulds and allowed to dry. The hardened material material was used to make soap bars.

2.7.2 Determination of total fatty matter (TFM)

The TFM was determined by petroleum spirit extraction. Soap (50 g) was dissolved in 500 ml warm water and transferred to a separating funnel. Three to four drops of methyl orange indicator were added, followed by 4N sulphuric acid until the indicator colour changed from orange to pink. Petroleum spirit (100 ml) was added and the separating funnel shaken vigorously for 30 s. The solution was then allowed to stand for a few minutes until the fatty

acid liberated soap formed a clear layer on top. The soap was skimmed off, washed with distilled water and dried to constant weight in an oven at 60⁰ C [11].

$$\% \text{TFM} = \frac{A-X}{W} \times 100$$

Where A= weight of wax + oil, X= weight of wax, W= weight of soap.

2.7.3 Determination of total alkali

The total alkali was determined by titrating excess acid contained in the aqueous phase with standard volumetric NaOH solution. Procedure reported in [12] was modified and used. One grams of finished soap was weighed and 5ml of ethanol was added to it. 0.5 milliliters of 1N H₂SO₄ solution was added to the mixture and heated till the soap sample dissolved. Test solution was titrated against 1 N NaOH using phenolphthalein as indicator. The total alkali was obtained with the formula;

$$\% \text{ Total alkali} = \frac{VA - VB}{W} \times 3.1$$

Where VA= Volume of acid W; VB= Volume of base; W= weight of soap.

2.7.4 Determination of % chloride

Five grams of finished soap was weighed and 50ml of distilled water added to it and heated to dissolve sample. The resulting solution was transferred into a 250ml volumetric flask and 10ml of 15 % Ca (NO₃)₂ was added to it and shaken to dissolve the soap. Distilled water was added to the solution to the 150 ml mark. The solution was filtered and methyl red was added to 50 ml of the filtrate was titrated against 10 N H₂SO₄ until a pink color was obtained. Resulting solution was titrated against 0.1 N AgNO₃ using K₂Cr₂O₇ as indicator, till a brick-red color was obtained [13].

$$\% \text{ Cl} = \frac{\text{Titre volume} \times 0.585}{\text{weight of soap}}$$

2.7.5 pH determination

The pH was determined using pH meter (827PH Metronm Model). A 5g of the soap shaving were weighed and dissolved with distilled water in a 100ml volumetric flask. The electrode of the pH meter was inserted into the solution of the soap and the pH reading was recorded. [14].

2.7.6 Foam ability test

A 2g of the soap was added to a 500 cm³ measuring cylinder containing 100 cm³ of distilled water. The mixture was shaken vigorously so as to generate foams. After shaking for some time, the cylinder was allowed to stand for 10 minutes. The height of the foam in the solution was measured and recorded [14].



Fig. 1. Shea nut fat (Hexane extract)



Fig. 2. Shea nut fat soap

Table 1. Some physical and chemical characteristics of fat extracted from shea nut

Parameters	Values
Color before/after solidification	Yellow / white
Physical state at room temperature	Solid
Oil yield (%)	38.74
Specific density (g/ cm ³)	0.96±0.03
Saponification value (mgKOH/g)	166.10±0.84
Iodine value (g I ₂ /100g)	53.54±0.39
Acid value (mgKOH/g)	14.26±0.33
Free fatty acid (% w/w)	4.20±0.04

Note: values are expressed as mean±standard deviation of triplicate determinations.

Table 2. Physicochemical characteristics of soap produced from shea nut fat

Parameters	Values/Observation
Ph	10.72
Foam height (cm ³)	31
Solubility in water	Slightly soluble
Texture	Soft
Color	Brown
%Total fatty matter	58±2.30
%Total alkali	0.57±0.08
% Chloride	1.26±0.01

Values are mean±standard deviation of triplicate determinations

3. DISCUSSION

The percentage yield of the oil from the shea nut kernel was 38.74% even though the value is lower than 48% reported for *Jatropha curcas* L. seed oil [14] is considered to be higher than 31.23% reported for Sponge Gourd (*Luffa cylindrica*) [15] all recommended for use in the preparation of soap. The specific density of the oil is 0.96g/cm³ which is within 0.915g/cm³ and 0.923g/cm³ reported for two varieties of *Sesamum indicum* L. seed oil [16], but is higher than 0.90 g/cm³ presented for garlic (*Allium sativum* L.) oil [17] and 0.89g/cm³ for African Oil Bean seeds *Pentaclethra macrophylla* [18]. This indicates that this oil could be used for commercial purposes.

The result of physicochemical analysis of (Table 1) showed that the shea oil (Fig. 1) has saponification value of 166.10mgKOH/g. This might have justify why the soap is hard, which is higher than Sponge Gourd (*Luffa cylindrica*) oil 112mgKOH/g [15]. Therefore, the value of saponification of the shea oil indicates its use in the soap industry. Higher saponification justifies the usage of fat or oil for soap production.

Iodine value of $53.54 \pm 0.39 \text{g I}_2/100\text{g}$ was obtained which is higher than $30.33 \pm 2.40 \text{g I}_2/100\text{g}$ for Sponge Gourd (*Luffa cylindrica*) oil [15]. Iodine value is an indication of double bonding in the molecular structure and influences the long term stability properties of oil which is important for storage. Iodine values below 100 are classified as non-drying fats.

Acid value is an important index of physicochemical property of oil which is used to indicate the quality, age, edibility and suitability of oil for use in industries such as paint [19]. The value 14.26 mgKOH/g was obtained for the shea oil which is higher than the 0.6 mgKOH/g) reported [20] for edible vegetable oil. Thus oil is more susceptible to lipase action.

The acid content of edible fats is given by the quantity of free fatty acids deriving from the hydrolytic rancidity of triglycerides. As this alteration occurs, in unsuitable conditions for the processing and preservation of fats, acidity represents a basic indicator of the genuineness of the product. The free fatty acid was found to be 4.20 ± 0.04 (% w/w). High concentration of free fatty acids is undesirable in crude oils because it result in large lose of oil during refining and can cut off flavors and shorten the shelf life oils [21]

The shea soap (Fig 2) was prepared by a product of saponification reaction between NaOH solution and oil extracted from shea kernel. "The prepared brown colored soap has a foam height of 31cm^3 and the soap forms a clear solution and slightly soluble in water".

Table 2 show the physicochemical characteristics of soap produced from shea nut fat.

The total fatty matter (TFM) of shea soap was reported to be (58.66 %) The difference in TFM is responsible for high moisture content and the kind of the used fatty material. Lower TFM values are due to presence of unreacted NaOH in the mixture [11]. However, dry skin needs soap which is high in TFM of 80%. This rehydrates the skin making it smooth and in addition the high oil content within the soap acts as lubricant throughout the day [22].

The determination of % chloride level in the soap is important as excess amount causes cracking of the soap. The % chloride value (1.26) reported in this present study is higher than (1.15) of % chloride of neem soap [23] which indicates that the value obtained is enough to sustain the soap and prevent it from cracking. Chlorinated water was used to dissolve NaOH pellet. This may be the reason for high chloride content of the soap.

Total alkalinity is the total alkaline materials present in the finished soap. This includes many alkaline components such as hydroxides, sodium (II) oxide, carbonates and bicarbonates. It involves the decomposition of soap by known volume of standard volumetric mineral acid solution, extraction and separation of liberated fatty matter [11]. The soap total alkali value was 0.57% which is higher than 0.24% of neem soap [23]. NaOH and NaCl contribute to the total alkali of shea soap.

From the result pH 10.7 of the shea soap is within the range of pH soap 9-11 [22]. This value is higher than cotton seed oil soap [10]. This high value is as a result of incomplete hydrolysis resulting from saponification process. It can be overcome by adding excess fat or

oil to reduce the harshness of soap [10]. The pH of this soap indicates that, the prepared soap is not corrosive to the skin. An alkaline substance neutralizes the body's protective acid mantle that acts as natural barrier against bacteria and viruses. Healthy skin has pH 5.4 – 5.9 [24].

4. CONCLUSION

The brown colored shea soap was produced from shea nut fat obtained by Soxhlet extraction and the physico-chemical profile exhibited by the shea oil makes it a possible raw material for soap making, cosmetics (creams and lotion). The properties exhibited by the soap indicates its suitability for commercial production and it could be used as a substitute for palm oil in producing cosmetic toilet soaps with favorable medicinal properties.

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COMPETING INTERESTS

Authors have declared that no competing interests exist.

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