EXTRACTION AND CHARACTERIZATION OF NIGERIA SHEA BUTTER OIL

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Abstract

The industrialization of oil seeds is fast becoming an important agro-industrial activity worldwide. This study presents the results of extraction, refining and characterization of both crude and refined Nigerian shea butter oil. The results of characterization of crude and refined oil shows specific gravity of 0.86 and 0.89, moisture content of 2.29 % and 0.12 %, saponification value of 389.89 mg KOH/g and 162.61 mg KOH/g, while the acid value of 8.42 mg KOH/g and 3.36 mg KOH/g, peroxide value of 15 meq/kg and 9.40 meq/kg, and refractive index of 1.472 and 1.467 respectively with it colour observed as milky-cream which are within the required standard specification. The grading of the oil based on the west African standard classified the crude and refined shea butter oils as 3rd and 2nd grade respectively, thus establishing their potential for the needs of food industry for manufacturing confectionary, chocolate, edible oil, and a basis for margarines. The shea butter of 3rd grade is recommended to be used in soap-making or further refined for direct consumption.

Keywords: Shea butter, Shea nut, Extraction, Vegetable oil

Introduction

In Nigeria, there are abundant vegetable oils, namely; palm oil, coconut oil, groundnut oil, rubber seed oil, cotton seed oil, olive oil, soya bean oil and conophor seed oil, e.t.c (Dawodu, 2009). Vegetable oils are normally extracted from fruits, seeds kernel and nuts either by mechanical press or by solvents (Akpabio *et. al.,* 2011).

Shea butter is a vegetable fat extracted from the kernel of the fruit of the shea tree (*Vitellaria paradoxa),* a tree belonging to the family of *sapotaceae.* The tree is the main indigenous oil producing wild plant spontaneously growing in Africa (Honfo *et al.,* 2012). Hee, (2011) reported that Shea tree begins to bear fruit of commercial quantities after approximately 20 to 50 years. In comparison to other trees grown as plantation crops, shea trees take much longer time to reach maturity, which discouraged its commercial plantation. Alander, (2004) reported that the trees do not reach maturity until 45 years and they can continuously produce shea nuts for up to 200 years in commercial quantities.

The tree grows wild across a 5000 km wide belt of savanna (Masters *et al*., 2004), including West African countries like Senegal, Mali, Côte D'ivoire, Burkina Faso, Togo, Ghana, Benin, Nigeria, Niger, Cameroon, and in East Africa such as Uganda, Sudan and Ethiopia (Hee, 2011). Four of these countries account for about 600,000 MT (app. 80 %) of world shea nut production which includes, Nigeria (370.000 MT), Mali (85.000 MT), Burkina Faso (70.000 MT) and Ghana (61.000 MT) (Karen, 2005). Among these countries, Ghana and Burkina Faso are the main shea nut exporters (Hee, 2011). Nigeria produces about 50 % of global shea nut production, but tends to consume most of its shea nuts locally (Karen, 2005). In Nigeria, the shea tree grows in Niger, Kwara, Kebbi, Kaduna and Oyo states. Shea butter oil extracted from shea nut is botanically called *Butyrospermum parkii*. It is a soft paste of melted fat with a milky colour in solid form and brownish when melted with a characteristic odour (Eka, 1997). It is an ancient African commodity that plays an important role in village life (Honfo *et al.,* 2012). This native source of edible oil or fat is traditionally used for frying, adding to sauces, as a skin pomade, for medicinal applications, to make soap, oil for lanterns and for cultural purposes at ceremonies, such as births, weddings and funerals. It can also serve as a

cocoa butter equivalent in the manufacture of chocolate as well as an ingredient in cosmetics (Alander, 2004).

This exceptionally rich vegetable extract contains fatty acids, phytosterol and unsaponifiable matter which stimulate the skin's natural renewal process (Asuquo *et al.,* 2010). It contains fatty acid triglyceride and a high amount of unsaponifiable matter, which ranges from 2.5 to 15 % (Eka, 1997). According to Tella (1979), shea butter oil contains cinnamic acid, a substance that helps protect the skin from harmful ultra-violet rays. Asuquo *et al.,* (2010) reported that unrefined shea butter oil is superior to refined shea butter oil in that it retains all its natural vitamins, especially vitamins A and E. It also has natural anti-oxidant properties due to its tocopherol content. Shea nut contains 37-55 % of fats; it is composed mainly of two fatty acids, stearic and oleic, which together account for 85-90 % of the total fatty acids (Ferris *et al.,* 2001; Maranz *et al*., 2004). Soft shea butter has high oleic content (Badifu, 1989). Honfo *et al*. (2012) reported that Shea butter oil contains 2-6 % palmitic acid (C16), 15-25 % stearic acid (C18), 60-70 % oleic acid (C18) and less than 1 % linoleic acid (C18) and 5-15 % linolenic acid.

Asuquo *et al.,* (2010) in their effort to fill the gap existing in literature on the Nigerian shea butter reported the extraction of shea butter from shea nut obtained from Karno, a satellite town in Abuja. The author reported that there are usually variation in the Physico-chemical composition of vegetable oils depending on environmental factors such as available rain- fall, soil fertility, maturation period, agronomic practices and genetic substitution. Sonau *et al.,* 2006 added that the composition of shea nut product depends on a number of criteria particularly the geographical occurrence, its botanical origin, handling of the seeds and processing e.g drying time, ripening.

This present study is aimed at extraction and characterization of a typical Nigerian shea butter oil with a view of placing it on the world map as the sample studied was obtained from the ancient city of Bida, a major producer of shea nut in Niger state of Nigeria.

Materials and Methods

Preparation of the Seed for Extraction: Shea butter (*Butyrospernum parkii*) seeds (nuts) were collected from Bida in Niger State, Nigeria. The seeds were dehulled, cleaned and dried under the sun for a day and later dried in the oven for three hours at 50 °C to ensure that moisture content was reduced to the bearest minimum.

Oil Extraction: The prepared seed were oven dried at 70 ^{O}C until a constant weight was obtained, then grinded into sizes. The extractor used was soxhlet apparatus with n- hexane as solvent. After extraction, the mixture of the solvent and extract was allowed to cool and then filtered to remove solid particles. The filtrate was concentrated under vacuum in a rotary evaporator (Akpan *et al.,* 2005). The results obtained were noted. The extracted oil was analyzed for the physical and chemical properties. All reagents used were of analytical grade.

Degumming and Purification: The oil was heated to 60 ^{0}C and activated carbon (commercially purchased) introduced into the heated oil, the oil became decolourized. The bleached oil was mixed with water thoroughly and heated again to 60 ^{O}C, stirred vigorously for 15 minutes, filtered, cooled and the sludge on the filter paper was discarded. The extracted oil (purified) was transferred into a glass bottle and stored in a refrigerator until all analyses were completed (Akpan *et al.,* 2005).

Physico-Chemical Characterization

pH Determination: Two grams (2 g) of the sample was poured into a clean dry 25 ml beaker and 13 ml of hot distilled water was added to the sample in the beaker and stirred slowly. It was then cooled in a coldwater bath to 25 °C. The pH electrode was standardized with buffer solution and the electrode immersed into the sample. The pH value was read and recorded (Akpan *et al.,* 2005).

Moisture Content Determination: Fifty grams (50 g) of the cleaned sample was weighed and dried in an oven at 80 °C. After every 2 hours, the sample was removed from the oven and placed in the desiccator for 30 minutes to cool. It was then removed and weighed (Akpan *et al.,* 2005). The percentage moisture in the seed was then calculated from:

$$Moisture = \frac{100(W_1 - W_2)}{W_1} \% \quad \text{..... 1}$$

where;
W_1 = Original weight of sample before drying (g), W_2 = Weight of sample after drying (g)

Specific Gravity Determination: The specific gravity bottle was cleaned with acetone, ether and dried in an oven at 60 ^{o} C. The weight of the empty bottle was taken, after which the bottle was filled with the oil sample and properly covered. The weight was then recorded using a weighing balance, after which the sample was removed from the bottle. The bottle was properly washed and filled with distilled water, after which the weight was taken and finally, the specific gravity was computed using the relationship below (Akpan *et al.,* 2005).

$$\text{Specific Gravity} = \frac{W_o - W}{W_1 - W} \quad \text{..... 2}$$

where, W = Weight of empty bottle (g), W_o = weight of the bottle and oil content (g), W_1 = Weight of bottle and water content (g).

Acid Value Determination: Two grams of the sample was dissolved in 50 cm^3 of mixed neutral solvent (25 cm^3 diethyl ether with 25 cm^3 ethanol carefully neutralized with 0.1M NaOH using 1% phenolphthalein solution). The mixture was titrated with 0.1M NaOH aqueous solution with constant shaking to faint pink colour (Akpan *et al.,* 2005).

$$Acid\ value = \frac{Titre\ value \times 5 \times 61 \times 0.00282}{Weight\ of\ sample\ (g)} = mgKOH/g \quad \text{..... 3}$$

Free Fatty Acid Determination: The amount of free fatty acid (FFA) was calculated as being equivalent to half the value of acid value (Akpan *et al.,* 2005), that is,

$$FFA = \frac{Acid\ value}{2} = mgKOH/g \quad \text{..... ..... 4}$$

Saponification Value Determination: 0.5 M KOH was prepared in 95 % ethanol, 2g of oil sample was weighed and 25 cm of KOH was added, 25 cm^3 of the blank solution was also measured into a conical flask. The two samples were then connected to a reflux apparatus and allowed to boil for an hour until the reflux is completed, 1 cm^3 of phenolphthalein was added to the mixture and the resulting mixture was titrated while hot against 0.5 M HCL acid solution. The volume of the acid used to attain the end point was recorded, the blank determination was carried out using the same procedure described above until the colour changes from blue to transparent white, then the volume of acid used was noted, the Saponification value was determined using the relationship below (Akpan *et al.,* 2005).

$$Saponification\ value, (S.V) = \frac{56.1 \times T(V_o - V_1)}{M} \quad \text{..... 5}$$

Where, T= Molarity of the standard KOH solution used (M), V_o = Volume of acid used for the first titration with oil sample (cm^3), V_1= Volume of acid used for the second titration of the blank solution (cm^3), M= Mass of the oil sample used (g).

Peroxide Value Determination: a known weight (2 g) of sample was weighed into clean dried boiling tube, 1 gram of potassium iodine (KI) powder was added to the oil and 20 cm^3 of the solvent mixture (i.e, glacia acetic acid and chloroform in the ratio 2:1). Then the boiling tube was placed in boiling water bath so that the liquid mixture boils within 30 seconds and allowed to boil vigorously for not more than 30 seconds, the content after boiling was quickly poured into a flask containing 20 cm^3 of 5 % potassium iodine (KI) solution and the tube was washed out twice with 25 cm^3 of water. Then the mixture was titrated with 0.002 M sodium thiosulphate using fresh 1 % starch solution, a blank titration was carried out at the sample time, the peroxide value was calculated using the relationship below (Akpan *et al.,* 2005).

$$Peroxide\ value = \frac{T \times M \times 1000}{weight\ of\ sample\ (g)} \quad \text{.....} \quad 6$$

where T = titre value of $Na_2S_2O_3$ = Sample titre – Blank titre, M = Molarity of $Na_2S_2O_3$

Refractive Index Determination: The refractive index was determined using Abbey refractometers. The glass prism of the refractometer was thoroughly cleaned with alcohol to ensure that it is free from dust, a drop of oil sample was placed on the lower prism and smeared, then closed with the other covering prism and the light source of the refractometers was switched on, while viewing through the telescope. The coarse adjustment knob was rotated until the black shadow appears central in the cross wire indicator and while still viewing through the telescope, the fine knob adjustment was made until the rainbow-coloured fringe which appeared on the black dividing line disappeared, the coarse knob was rotated to give fine adjustment and make the black shadow appear exactly central in the cross wire indicator. The reading under the telescope and that of the fine adjustment knob were noted and divided by 10,000, this value was then added to the value obtained through the telescope to give the value of the refractive index of the oil at room temperature (Akpan *et al.,* 2005).

Results and Discussions

Results obtained on the qualities of the oil obtained from shea nut oil are presented in Table 1 while Table 2 represents the qualities of different grades of unrefined shea butter.

Table 1: Characteristic of unrefined and refined shea butter

Characteristics	Required	Crude oil	Refined oil
Saponification value (mg KOH/g)	180 – 360	389.89	162.61
pH	3.18 ± 0.83	4.38	4.58
Free fatty acid (%)	1.1 – 3	4.21	1.68
Acid value (mg KOH/g)	2.30 ± 1.6	8.42	3.36
Specific gravity	0.8 – 1.0	0.86	0.89
Peroxide value (Meq/kg)		15	9.40
Moisture content (%)	0.06 – 0.2	2.29	0.12
Refractive index		1.472	1.467

Grading of Shea Butter Oil

The West African regional standard classified shea butter oil into three grades; Grade 1 are classified as those that can be use by cosmetic and pharmaceutical industries and for direct consumption, Grade 2 shea butter oil are employed in food industries for manufacturing confectionaries, chocolate, edible oil and for making margarine, Grade 3 are used in making soap and can further be refined for direct consumption.

Table 2: Quality characteristics and grades of unrefined shea butter

Parameters	Grade 1 Min – Max	Grade 2 Min – Max	Grade 3 Min – Max
Moisture content (%)	0 – 0.05	> 0.05 – 0.2	> 0.2 – 2.0
Free fatty acid (%)	0 – 1.0	> 1.0 – 3.0	> 3.0 – 8.0
Peroxide value (meq/kg)	0 – 10.0	>10.0 – 15.0	>15.0 – 50.0
Insoluble impurities (%)	0 – 0.09	>0.09 – 0.2	> 0.2 – 2.0

Source: Regional Technical Committee, 2006

Moisture is a chemical contaminant which is usually well mixed with oil. Presence of moisture in oil affects the quality of the oil, it has been reported that significant amount of moisture in oil support microbial growth (Alirezali *et al.,* 2011). Results obtained on the moisture contents of crude and refined oil as presented in Table 1 indicate that the moisture contents of shea butter oil were 2.29

and 0.12 % for crude and refined oils respectively. The value of the moisture in refined oil shows appreciable decrease with respect to the standard of 0.06 – 0.2 % as reported by Okulle *et al*. (2000). However, a lower value of 0.1 % was reported by Asuquo *et al*. (2010) though the value was higher than 0.037 % obtained by Enweremadu and Alamu, (2010) while the crude has relatively high moisture content when compared to the refined. The difference observed could be due to the age of the oil or the method of extraction employed. Based on the classification of shea butter, the moisture content of the crude oil falls above the 3rd Grade and the refined within the 2nd Grade. Therefore, refining the crude oil improves its quality attributes.

Specific gravity is an important physical property that can give information on the identity of the sample as well as aid in detection of adulteration of Shea butter oil (Hee, 2011). It can also provide information for the shippers on the weight of the Shea butter from the given volume while exporting it in large volumes (Hamilton and Rossell, 1986). The specific gravity for this oil was determined to be 0.86 and 0.89 for crude and refined oil respectively. These values show close proximity to 0.8 – 1.0 as the required standard. Also presented in Table 2 is the refractive index of the oil which can be used to establish the purity of fats and oils when suspected to be adulterated (Olaniyan and Oje, 2007) as well as one of the important physical characteristics for identification of oils and fats. In this study, refractive index was measured to be 1.472 and 1.467 for crude and refine oil respectively. After refining, there was a negligible decrease in refractive index which implies that this oil still falls within where the refine oil falls within the range of a typical refractive index of Shea nut oil, 1.463 – 1.467 as reported by Hamilton and Rossell, (1986). These values are consistent with the refractive index of *chrysophyllum albidum* that falls between 1.45 – 1.47 as reported by Ochigbo and Paiko (2011). Asuquo *et al.* (2010) reported 1.47 for Castor oil and 1.46 for rubber seed oil.

Peroxide value is a valuable measure of oil quality as it provides an indication to the stability of the oil and the level of deterioration of fats. The oil under study has a peroxide value of 15 and 9.4 Meq/kg for crude and refined oil respectively. This low peroxide value showed that the oil cannot easily go rancid. It is important to add that the value obtained from this study for refined oil is lower than 14.2 and 12.5 reported by Asuquo *et al.,* (2010) and Eweremadu and Alamu, (2009) respective but shows a close proximity to the value obtained for crude. The differences in values strongly suggest that factors such as rainfall, soil fertility, maturation period, agronomic practices and genetic substitution may have contributed to these differences (Sonau *et al.,* 2006).

Peroxide value as one of the classification parameters shows the value obtained for the crude fall within the 3rd Grade and that for the refined to be a 2nd Grade.

Saponification value is the number of milligram of potassium hydroxide required to neutralize the fatty acid present in a hydrolysis reaction. The high saponification value of 389.89 and 162.26 mg KOH/g for crude and refined shea butter oil is an indication that the Shea butter oil is a normal triglyceride molecule. The higher the saponification value of the oil, the higher is the lauric acid content of that oil. The lauric acid content and the saponification value of the oil serve as important parameters in determining the suitability of the oil in soap making (Asuquo *et al.,* 2010). Also the acid value of the Shea butter oil was found to be 8.42 and 3.36 mg KOH/g for crude and refined oil respectively and a corresponding free fatty acid value of 4.21 % and 1.68 %. This is in close agreement with the work of Enweremadu and Alamu, (2009) who obtained a value of 3.62 mg KOH/g as acid value. Therefore the Grading system shows the free fatty acid of the crude and refined oil to fall within the 3rd and 2nd Grading respectively.

Conclusion

Nigeria is in the fore front of shea nut production and the market for this product is expanding from domestic consumption to industrial feedstock for production of variety of valuable end products and as a potential source of economic development in rural area where the tree are widely grown. The results from this work show that the refined oil has a low moisture content establishing the fact that the oil is not susceptible to rancidity under storage for a longer period of time and the corresponding low peroxide value clearly attest to this fact. The low acid and free fatty acid value are good indicators that the oil can be used as a feedstock for biodiesel production as the values

obtained are close to threshold limit for the production of biodiesel. The refined oil can be classified as 2nd Grade which is used as raw materials in food industries while the crude to fall within the 3rd Grade which can be used in soap making and can be further purified to the 2nd grade. This classification was based on the West African regional standard.

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