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Soap preparation from Soxhlet extracted Nigerian Cotton seed oil

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ABSTRACT

The large scale industrial production of cotton seed oil in Nigeria has unrestricted its use for only food application. A journey has started towards the utilization of this seed oil for soap production. Physicochemical analysis of the extracted seed oil was carried out in order to justify its usefulness in soap industry. The following values were obtained for the various parameters measured; Saponification value 199.42 ± 0.53 mgKOH/g, Iodine value 119.78 ± 0.81g I_2/100g and Acid value 0.81± 0.01mgKOH/g. The oil yield was 48%. The analytical values obtained were significantly in favour of the utilization of the indigenous cotton seed for soap production on commercial scale. The pH of the soap was 9.38, comparably within the higher pH range of 9-11 set by the National *Agency for Food and Drug Administration and Control (NAFDAC), mostly due to incomplete alkali hydrolysis resulting from the saponification process. The foam height of the soap was 4.5cm 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: Cotton seed oil, solvent extraction, soap, pH, foamability.

INTRODUCTION

Cottonseed oil is obtained from the seed of cotton plant. The cotton plant (*Gossypium hirsutum* or G. b*arbadenseis*) is grown for its fibre. The oil is a by-product with about 12 per cent of the gross value of the total product. Cottonseed oil was once the major vegetable oil competing with the more widely-used animal fats. Today, it occupies ninth place in production tables after five vegetable oils (soybean, palm, rape/canola, sunflower and groundnut) and three land animal fats (tallow, lard and butter) [7].Cottonseed oil is unusual among commodity vegetable oils in that it contains a relatively high level of palmitic acid (typically 23%) along with oleic acid (17%) and linoleic acid (56%).[14]. Cottonseed oil is a member of a particularly useful group of vegetable oils, whose fatty acids consist substantially of C-16 and C-18 fatty acids containing no more than

two double bonds. Its fatty acid profile is typical of the oleic/linoleic group of vegetable oils, as these two fatty acids make up almost 75% of the total. Fatty acid composition of cotton seed oil [7]. Palmitic, the major saturated fatty acid in cottonseed oil, has been identified as a β′promoter when it is in the *sn*-1 or *sn*-3 positions, therefore, it is stable in the β′crystal form, which is desirable in many products because this stability promotes a smooth, workable consistency, usually referred to as plasticity [7]. The distribution of the fatty acids in cottonseed oil is considered to be nonrandom, with the saturated fatty acids positioned predominantly in the *sn*-1 or *sn*-3 positions and the unsaturated fatty acids in the *sn*-2 position. Because linoleic, oleic, and palmitic fatty acids account for over 90% of the fatty acid composition of cottonseed oil, most of the triglycerides contain some combination of these fatty acids. Analysis of cottonseed oil by semiquantitative thin-layer chromatography indicated that the distribution of saturated (S) and unsaturated (U) fatty acids in the *sn*-1, 2, and 3 acyl positions were 11.8% SUS, 4.4% SSU, 12.3% USU, and 42% UUS [17].Almost 30% of the triglycerides contain only unsaturated fatty acids, but no molecules are completely saturated [17]. Cottonseed oil was one of the earliest oils to be extracted from the seed. Food applications have been a major use for cottonseed oil but it has also been used in soap, lubricants, sulfonated oil, pharmaceuticals, protective coastings, rubber, as a carrier for nickel catalysts, and, to a lesser degree, in the manufacture of leather, textiles, printing ink, polishes, synthetic plastics, and Resins [8]. This research work is aimed at extraction of oil from cotton seed using soxhlet apparatus and n-hexane solvent, the objective was to utilize the extracted seed oil for soap production.

MATERIALS AND METHODS

Seed material

The cotton seeds were collected from Inter-ginnery plant at Gusau in Zamfara state, Nigeria. The shells were cracked with sharp knife to remove the seed, which were grounded into small sizes and then sundried for 5days.

Oil extraction

The oil was extracted by soxhlet extraction method using n-hexane at 60ºC, which was then refluxed at 70 ºC to remove excess solvent used in the oil. The crude cotton seed was clarified using 0.5M NaOH [11].

Chemical Analysis

The chemical analysis of the oils was carried out using the methods reported [5], [1] and [6].

Saponification value: 2 g of the oil sample was added to a flask with 30cm^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 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: 0.4 g of the sample was weighed into a conical flask and 20cm^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, 20 cm^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 [1].

The iodine value (I.V) is given by the expression

$$IV = \frac{12.69C\ (V_1-V_2)}{M}$$

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 cm^3 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.

Saponification Procedure
70cm^3 of 170g/dm^3 alkali solution was poured directly into the beaker containing the fat and oils in the ratio 1:1(v/v). The fats/oil was warmed gently and was poured into the beaker followed by the alkali solution to form an intimate mix and then stirred frequently for 10-15 minutes using stirring rod. The saponification mixture was then poured into moulds. After pouring, the soap was allowed to harden by air-drying for 24hours and was then tested for lathering and cleaning [24].

pH Determination:
The pH was determined using a pH meter (827 pH lab Model). 10g of the soap shavings was weighed and dissolved in distilled water in a 100cm^3 volumetric flask. This was made up to prepare 10% soap solution in line with literature report [12].The electrode of the pH meter was

inserted into the solution. The pH reading was recorded. The steps were repeated using soaps produced from each fat or oil.

Foam ability Tests

About 2.0g each of soap (shavings) was added to a 500cm^3 measuring cylinder containing 100cm^3 of distilled water as reported [16] for synthetic detergent. 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. The steps were repeated using soaps produced from each fat or oil.

RESULTS AND DISCUSSION

Table 1: Physicochemical characteristics of the Nigerian cotton seed oil

Parameter	Values
Saponification value mgKOH/g	199.42 ± 0.53
Iodine value g I_2/100g	119.78 ± 0.81
Acid value mgKOH/g	0.81± 0.01
Oil yield (%)	48
Physical state at room temperature	Liquid

The values are mean and standard deviation of triplicates determination.

Table 2: Physical and chemical characteristics of the Nigerian cotton seed oil soap.

Parameter	Results
pH	9.38
Foam height(cm)	4.50
Colour of soap solution	White
Solubility in water	Slightly soluble

The values are mean of triplicates determinations.

Table 3: pH of the various soap samples

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

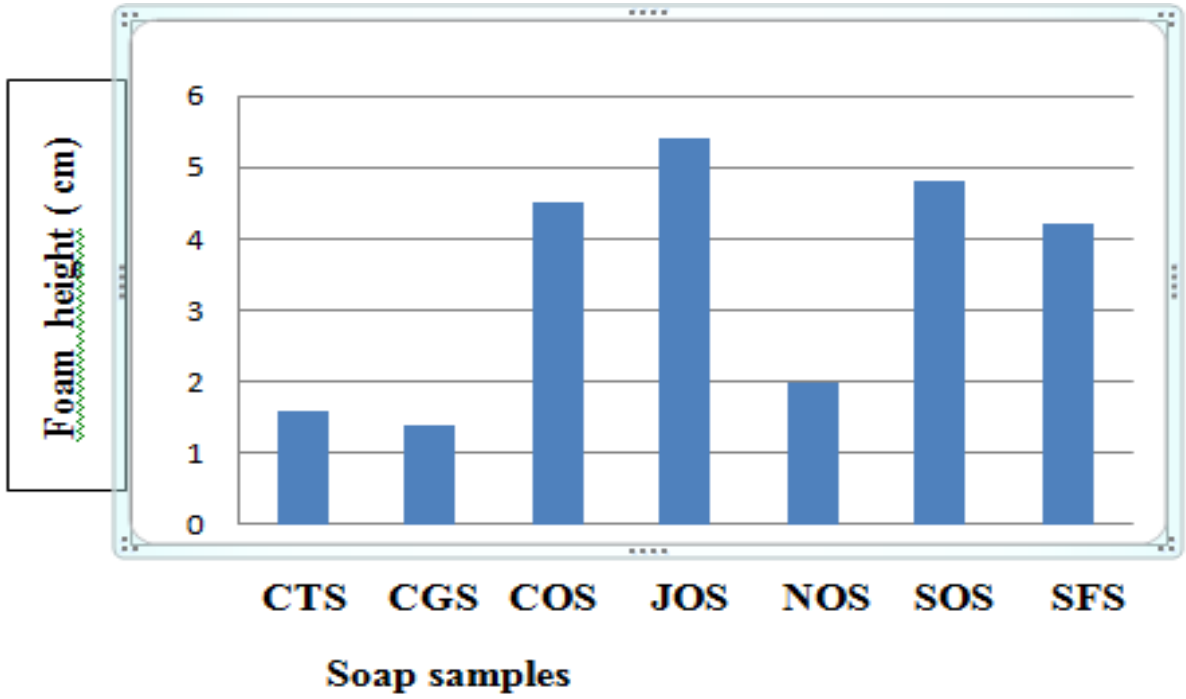
The values are mean of triplicates determinations.

Table 4: Foam ability of the various soap samples.

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

The values are mean of triplicates determinations.

Fig.1. Representation of foam ability as a function of foam height of the various soap samples.



CTS = Castor seed oil soap
CGS = Castor glycerine soap
COS = Cotton oil soap
JOS = Jatropha oil soap
NOS = Neem oil soap
SOS = Sesame oil soap
SFS = Shea nut fat soap

DISCUSSION

The results of the physicochemical analysis (**Table 1**) showed that Nigerian cotton seed oil has Saponification value of 199.42 ± 0.53 mgKOH/g which is lower than 213mgKOH/g in neem seed oil [2] and 253.2mgKOH/g in coconut oil [10] higher than that of *Dennettia tripatala* fruit oil(Pepper fruit)159.33±1-20 [18] and African pear oil 143.76 mgKOH/g which could be good for soap making [15] 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 119.78 ± 0.81g I_2/100g was obtained which is higher than 104.3g I_2/100g for sesame seed oil [22] and 84.8 g I_2/100g for ground nut oil [23] useful in the manufacture of soaps and lower than the Iodine value of *C. lanatus* 119.8 g I_2/100g [3] which is within a range of semi-drying oils consisting predominately polyunsaturated fatty acids mainly oleic and lenoliec fatty acids. This class of oils whose iodine value is between 100 – 150 possesses the property of absorbing oxygen on exposure to the atmosphere; though do not do so sufficiently to

qualify them as drying oils. They become thicken and remain sticky but do not form a hard dry film. They are used in the production of margarine and soap [9]; [4].

An Acid value of 0.81 ± 0.01 was obtained which is lower than that of olive oil 17 mgKOH/g [13] and shea nut fat 10.49mgKOH/g reported [20] and 10.3mgKOH/g for shea nut butter reported [25] suitable for soap making.

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The pH of the prepared soap was 9.38 , 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) [20] 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. [18]

The foam height of the soap was 4.50 cm lower than that of Jatropha oil soap (5.4cm) and that of sesame oil soap (4.8cm) and higher than that of sheanut fat soap(4.2cm), cotton seed oil soap(4.5cm), castor glycerine soap(1.4cm) and Neem oil soap (2.0cm). The soap was milky in colour and slightly soluble in distilled water.

CONCLUSION

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

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