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SOAP PREPARATION FROM MECHANICALLY COLD PRESSED NIGERIAN NEEM (*AZADIRACTA INDICA*) SEED OIL

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ABSTRACT

Neem seed oil from the neem tree (Azadiracta indica) finds wide usage one of which is its utilization for cosmetics particularly soap products. The chemical analysis of seed oil was carried out using the methods reported by AOAC (1998), Akpan et al., (2006) and Bassir, (1978) which revealed that it had saponification, iodine and acid values of 148.8 ± 1.168 mgKOH/g , 73.76 ± 0.397 I_2/100g and 22.37 ±1.168 mgKOH/g respectively. The analytical values obtained were in favour of the utilization of the indigenous neem seed oil in soap production. The pH of the soap was 9.90, comparably within the higher 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 found to be 2.0 cm which is lower than that of Jatropha oil soap(5.4cm), Sesame oil soap(4.8cm), Cotton seed oil soap(4.5cm) and shea nut soap(4.2cm),t higher than that of Castor oil soap(1.6cm) and Castor glycerine soap(1.4cm). The soap was milk in colour and slightly soluble in distilled water.

***Keywords*:** Neem oil, Quality control, Saponification, Alkali free, Foamability.

INTRODUCTION

The *Azadiracta indica* tree, a member of the *Meliaceae* family is a native to the seasonally dry, tropical woodlands of North-east India and perhaps parts of Asia.(Csurhes,2008).

Neem tree is commonly found in towns and villages in the Northern part of Nigeria. It is mostly planted in large numbers along road sides.

Mechanical extraction is the most widely used method to extract neem oil from neem seed.(Liauw *et al.,*2008). Oil extracted from its seeds is composed primarily of triacylglycerols of oleic, strearic, linoleic, and palmitic acids. The seeds yield 40% of a deep yellow oil, well- known as 'Margosa oil' (Girish and Shankara Bhat, 2008). Of all other industrial uses of neem seed oil, like its use as lubricant and in pharmaceutical preparations like emulsions, ointments, poultices, and liniments, in India, it has been a major ingredient in soaps (National Research Council, 1992). This work is aimed at exploiting the use of the neem seed oil for soap preparation based on laboratory work to justify its said utilization.

MATERIALS AND METHODS

Neem seed oil material

The indigenous Neem Seed oil was obtained from Saberg International Ltd. producers of proudly mechanically cold pressed Nigerian Naija Neems (Neem oil) at Technology Incubation Centre, Birnin Kebbi, Kebbi State , Nigeria.

Chemical Analysis

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

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, 1 cm^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 25cm^3 of Dam's reagent was added to the flask using a safety pipette in fume chamber. A 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 (Akpan *et al.,* 2006)

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 cm^3 of neutral ethyl alcohol was heated with 10 g of oil or fat sample in a 250cm^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 cm^3. N/10 KOH used.

Preparation of alkali solution
The alkali solution(AnalaR Grade) used for the soap preparation was prepared by dissolving 170g of sodium hydroxide pellets in 1dm^3 volumetric flask and the volume made to the mark with water.

Saponification Procedure
For each soap formulation, 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 to obtain the soap bars.

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 100ml 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 as 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), which for long has been accepted method for measuring foaming performance. 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 found 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 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

Table 1: Physicochemical characteristics of the Nigerian Neem seed oil

Parameter	
Saponification value mgKOH/g	148.8 ± 1.168
Iodine value I_2/100g	73.76 ± 0.397
Acid value mgKOH/g	22.37 ± 1.168
Physical state at room temperature	Liquid

The values are mean and standard deviation of triplicate determinations.

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

Parameter	
pH	9.90
Foam height (cm)	2.0
Colour of soap solution	Milky
Solubility in water	Slightly soluble

The values are mean of triplicate determination

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 triplicates determination.

DISCUSSION

The results of the physicochemical analysis (**Table 1**)shows that Neem seed oil has Saponification value of 148.8 ± 1.168 mgKOH/g which is lower than that of *Dennettia tripatala* fruit oil(Pepper fruit)159.33±1.20 suitable for soap making (Nwinuka, and Nwiloh, 2009) but higher than that of African pear oil 143.76 mgKOH/g which could be good for soap making (Ikhuoria and Maliki, 2007). 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 73.76 ± 0.397 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 22.37 ± 1.168 mgKOH/g was obtained which is higher than that of olive oil 17 mgKOH/g (Davine and Williams,1961) and shea nut fat 10.49mgKOH/g reported by Oyedele (2002), which signifies a maximum purity and makes it suitable for soap production.

For the prepared soap the pH was 9.90 , within the higher pH range of 9-11 but 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) 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 resulted in the better quality of soaps that were free of cracks.(Kuntom, *et al*, 1999).The foam height of the soap was 2.0 cm lower than that of Jatropha, sesame ,sheanut fat and cotton seed soaps analyzed higher than that of castor and castor glycerine soap. The soap was milky in colour and slightly soluble in 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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