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Short Communication

SOXHLET EXTRACTION, PHYSICOCHEMICAL ANALYSIS AND COLD PROCESS SAPONIFICATION OF NIGERIAN *JATROPHA CURCAS* L. SEED OIL

*Warra, AA[1], Wawata, IG[2], Umar, RA[3] and Gunu, SY[1]
[1]Department of Biochemistry, Kebbi State University of Science & Technology, PMB 1144, Aliero, Nigeria
[2]Department of Pure & Applied Chemistry, Kebbi State University of Science & Tech., PMB 1144, Aliero, Nigeria
[3]Department of Biochemistry, Usmanu Danfodiyo University, PMB 2346, Sokoto, Nigeria

ABSTRACT

In search of oils that can replace edible oils used for soap making, *Jatropha curcas* L. seed oil was exploited. The lipid was extracted using n- hexane and analyzed for chemical properties. The parameters analyzed were Acid value, (1.20 ± 0.065mgKOH/g) Iodine value, (73.46 ± 5.00g I_2/100g) and saponification value (122.49 ± 2.59mgKOH/g). The percentage oil yield was 48. The lipid was used to prepare soap. The pH of the soap was 9.11, 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 5.4 cm and was higher than that of all other soap solutions analyzed. The soap forms a clear solution and was slightly soluble in distilled water.

Keywords: Jatropha oil, extraction, saponification, physicochemical analysis.

INTRODUCTION

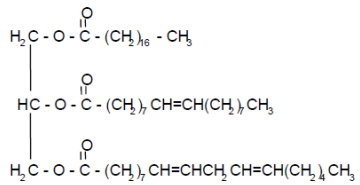
Jatropha curcas (Linnaeus) is a multipurpose bush/small tree belonging to the family of *Euphorbiaceae*. It is a native of tropical America, but now thrives in many parts of the tropics and sub tropics in Africa and Asia. Although most of the *Jatropha* species are native to the new world, approximately 66 species are native to the Old World (Heller, 1996). It is a tropical plant that that can be grown in low to high rainfall areas (Openshaw, 2000). It is considered as a potential source of non-edible fuel producing plant along with its different medicinal properties (Verma and Gaur, 2009). In Nigeria it is known as "binidazugu/cinidazugu" and 'lapa lapa" in Hausa and Yoruba languages respectively (Blench, 2007; Blench, 2003). *J. curcas* is being explored for its oil yield potential throughout the world (Ginwal *et al.*, 2004). Various methods for recovering this oil from the seeds have been investigated. Shah *et al.,* (2005) reported the use of combination of ultrasonication and aqueous enzymatic oil extraction method. Extracting oil from Jatropha seeds can be done either with a manual press, such as the ram press, or with a mechanical press, such as the oil expeller. With mechanic expellers (like sundhara press) up to 75-80% of the oil can be extracted. With hand press like the Bielenberg ram press only 60-65% of the oil can be extracted (5kg of seeds give about 1litre of oil (Henning, 2003). Extraction with organic solvents and water has been the main approaches. For research purposes, Sayyar *et al.* (2009) in their research on optimization and kinetics in the extraction of oil from *Jatropha* found Hexane to be the best solvent for the process as compared to petroleum ether. High oil content of *Jatropha Curcas* indicated that the oil is suitable as non-edible vegetable oil feedstock in oleochemical industries (biodiesel, fatty acids, soap, fatty nitrogenous derivatives, surfactants and detergents (Akbar *et al.*, 2009).

Due to its toxicity consequent upon presence of curcin and phorbol esters (King *et al.*, 2009) *J. curcas* oil is not edible and is traditionally used for manufacturing soap and medicinal applications (Jongschaap *et al.*, 2007). Goel *et al.* (2007) suggests that the detoxification or complete removal of phorbol esters is essential before its use in industrial or medicinal applications. The major toxin phorbol ester is not vulnerable to heat, but can be hydrolyzed to less toxic substances extractable by either water or ethanol (Usman *et al.*, 2009). The use of Jatropha in soap industry, (alternative Karitee Butter) and cosmetics is regarded as one of its non-energy use (Rijssenbeek, 2007).

Chemical structure

Jatropha seed oil chemically consists of triacylglycerol with linear fatty acid chain (unbranched) with/without double bonds.

*Corresponding author email: aliyuwarra@yahoo.com



This work is aimed at preparation of soap from soxhlet extracted Nigerian *Jatropha curcas* L. seed oil.

MATERIALS AND METHODS

Seed material

Indigenous *J. Curcas* L. seeds were obtained from *Jatropha Curcas* plant in a test plot in Warra town Ngaski local government area of Kebbi State, Nigeria. The plant was identified and authenticated by a Botanist at the Biological Sciences Department, Bayero University, Kano (BUK) Nigeria. Confirmation of taxonomic identity of the plant was achieved by comparison with voucher specimen (voucher No. 110) kept at the Herbarium of the Department of Biological Sciences. The seeds were selected and damaged ones were discarded. The seeds were cleaned, de-shelled and well dried and ground using laboratory plastic pestle and Mortar prior to extraction.

Oil extraction

The extraction of 5.0g of the grounded seed kernels was conducted in a soxhlet extractor using n-hexane (boiling point of 40–60ºC) for 6hours. The lipid was obtained after the solvent was removed under reduced temperature and pressure and refluxing at 70ºC to remove excess solvent used in the oil. Extracted seed oil was stored in freezer at −2ºC for subsequent physicochemical analyses.

Oil Yield

The oil, which was recovered by complete distilling of most of the solvent on a heating mantle was then transferred to measuring cylinder. The measuring cylinder is then placed over water bath for complete evaporation of solvent for about 2-3hours in accordance with the method reported by Pant *et al.* (2006) and volume of the oil was recorded and expressed as oil content (%) as follows:

$$\text{Oil content (\%)} = \frac{\text{Oil weight}}{\text{Sample weight}} \times 100$$

Chemical Analysis

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

Saponification value: About 2g of the oil sample was added to a flask with 30cm^3 of ethanolic KOH and was then attached to a condenser for 30minutes to ensure the sample was fully dissolved. After sample has cooled, 1cm^3 of phenolphthalein was added and titrated with 0.5M HCl until a pink endpoint was 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: 0.4g of the sample was weighed into a conical flask and 20cm^3 of carbon tetra chloride were added to dissolve the oil. Then 25cm^3 of Dam's reagent were 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 2hours 30minutes. 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 solution until the yellow colour almost disappeared.

Few drops of 1% starch indicator were 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) was obtained from 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: 100cm^3 of neutral ethyl alcohol were 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

For each soap formulation 70cm^3 of 170g/dm^3 alkali solution were 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 10-15minutes 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 were 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 electrode of the pH meter was inserted into the solution and the pH reading was recorded. The steps were repeated using soaps produced from each fat or oil.

Foam ability Tests

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 distilled water. The mixture was shaken vigorously so as to generate foams. After shaking for about 2minutes, the cylinder was allowed to stand for about 10minutes. 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

The results obtained are presented in tables 1 to 4.

Table 1. Physicochemical characteristics of *J. curcas L* seed oil.

Parameter	Observation
Saponification value mgKOH/g	122.49 ± 2.591
Iodine value gI_2/100g	73.46 ± 5.00
Acid value mgKOH/g	1.20 ± 0.065
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 prepared *Jatropha* soap.

Parameter	Observation
pH	10.11
Foam height (cm)	5.4
Color of soap solution	Clear solution
Solubility in water	Highly Soluble

The values are mean of triplicates determinations.

Table 3. pH of the various soap samples compared with *J. curcas* seed oil soap.

Soap sample	pH value
Castor oil based soap	9.70
Castor glycerine soap	9.60
Cotton oil soap	9.38
Jatropha oil based 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 as a function of foam height of the various soap samples compared with *J. curcas* seed oil soap.

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 determinations.

DISCUSSION

The physicochemical analysis (Table 1), determined for the soxhlet extracted indigenous Jatropha seed oil includes; Saponification value of 122.49 ± 2.591 mgKOH/g the value obtained was 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 beeswax (93 mgKOH/g), which are commonly 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 justifies the usage of fat or oil for soap production.

Iodine value of 50.50 ± 8.023 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 17mgKOH/g (Davine and Williams, 1961) higher than the 10.49 3mgKOH/g reported by Oyedele (2002), which signifies a maximum purity and made it suitable for soap production. For the prepared soap the pH was 9.11 (Table 3) 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 5.4cm (Table 4) higher than that of all other soap solutions analysed. The soap forms a clear solution and was and slightly soluble in distilled water. 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.

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. Hence even if the current boom for Jatropha production is based mainly on the incentive of producing biofuel, other possible range of products can be derived from Jatropha seed oil such as soap as demonstrated by this present research, which appears to be the first reported work in Nigeria.

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