

Suitability of sulphated *Jatropha curcas* seed oil as fatliquor for processed leather

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ABSTRACT

Jatropha curcas oil was synthesized to fat liquor and used in comparison with commercial fat liquor to process goat skins according to leather specifications in a tannery. The raw oil was characterized for the following physicochemical properties: pH - 5.2, Density - 0.92 g/cm³, Iodine value - 72.12, Saponification value - 224.40 mgKOH/g, Acid value - 13.2 mgKOH/g, Free fatty acid- 6.6, Specific gravity- 0.9 and Moisture content- 8%. The fatliquor was synthesized via a sulphation process and was characterized as follows: Iodine value - 60.00, Saponification value - 230.2 mgKOH/g, Acid value - 22.4 mgKOH/g, Free fatty acid- 11.2, Specific gravity- 1.2 and Moisture content- 20 %. After both synthesized fatliquor (SF) and commercial fat liquor (CF) were employed in the fatliquoring process, the fatliquored leathers were analyzed for several physicochemical properties namely: Tensile strength: CF -24.41 N/ SF-30.00 N; Percentage Elongation: CF-21 % /SF-60 %; Water absorption: CF-18.31 % /SF-18.33 %; Abrasion resistance: CF-2.63 % /SF-11.76 %; Shrinkage Temperature: 96 °C for both CF and SF; Displacement: CF- 9.2 mm/ SF-12.36 mm; Resistance to Compression: CF-2.62 kgf cm⁻²/ SF- 2.88 kgf cm⁻²; Thickness: CF-1.37 mm /SF- 1.42 mm and Indentation Index of 23 for both CF and SF. It is thus concluded that fat liquor from *Jatropha curcas* seed oil can serve as a sustainable replacement for commercial fat liquors in the tanning industry and will yield more desirable physico-mechanical properties of the leather.

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INTRODUCTION

Fatliquor constitutes a substantial raw material in the chemical finishing process of leather. Skins and hides lose large quantities of water and fats during the leather tanning process. This necessitates the lubrication of the fibres of tanned skins in order to impart desirable physical and mechanical properties like softness, elasticity, water repellency, elongation at break and tensile strength properties [1]. The inclusion of fatliquor (oil-in-water emulsion) into leather is often the last of the wet processing stages of leather manufacture and lubricates the leather fibres preventing this sticking and producing softer leather. Fatliquors keep the fibres apart during drying and help to reduce frictional forces within the fibres' weaves [2]. Fatliquors are introduced

into the leather matrix in finely dispersed form which breaks the interfacial tensions between the oil and water which makes them miscible [3]. This emulsification process can be achieved through the introduction of sulphonate groups into the leather structure, through addition of surfactants to the composition of the fat liquor, or by exposing natural and sulphated fats to ultrasonic waves. Due to overdependence on natural reserves, the use of renewable oil sources to synthesize fat liquoring agents is in present demand. Some oils commonly used in synthesizing fatliquors include sperm whale oil, jojoba oil, cod oil, linseed, cotton seed, neat foot, olive oils. Anionic fatliquors are usually employed for fatliquoring chrome tanned leathers and are generally prepared by sulphation, sulphonation or bi-sulphitation of fats and oils [4].

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Sulphation is a process of rendering oil soluble for stable emulsion with various chemicals by reacting it with sulphuric acid to form sulphated oil, which will possess a negative or anionic charge when emulsified. Sulphated oils (RCHOSO₃NaR') are generally synthesized by adding concentrated sulphuric acid to the oil at a temperature below 25 °C [5]. There is a dire need for fatliquors synthesized from non-edible oils of indigenous sources which are renewable and sustainable as against imported synthetic fat liquors. *Jatropha curcas* also known as the French physic nut tree is a drought-resistant perennial tree that tends well in soils with low nutrient content under arid and semi-arid conditions [6]. The tree takes origin from Brazil but has since spread to other parts of the world. In Nigeria, it is known as "yayan mamulu" among the Hausa people and "lakpa - lakpa" among the Yoruba people. It reaches heights of up to five metres and has advantages such as rapid growth, high seed productivity, suitability in tropical and subtropical regions of the world. *Jatropha curcas* has oil content of as high as 64.4 % in Nigeria [7] but on the average between 38.7 % to 45.8% [8] worldwide and does not compete with food demand. With each mature tree producing between 5-15 kg of seeds three times a year, a harvest of 5,000 kg of seeds per hectare per year, can give 1,500 litres of oil annually. The oil is chiefly employed in biodiesel production and in the production of soaps. This study was aimed at synthesizing fat liquor from *Jatropha curcas* seed oil, characterizing the fat liquor produced as against the raw oil, comparing the fatliquor produced and selected commercial conventional fatliquor to process chrome tanned leather; and finally carrying out physicochemical tests on the leathers processed with both fatliquors. The results

MATERIALS AND METHODS

The *Jatropha curcas* raw oil was sourced from National Research Institute for Chemical Technology, Zaria, Kaduna State, Nigeria and the Commercial fatliquor from Kano, Nigeria.

Chemical Analysis of Raw *Jatropha curcas* Oil

Several physicochemical properties such as determination of saponification value,

iodine value, acid value, free fatty acid value, specific gravity and moisture content, were analysed for in raw *Jatropha curcas* oil to ascertain its suitability for synthesis into a fatliquor to be used in leather processing.

Determination of Saponification Value

The method was described in AOAC [9]. The saponification value was determined by dissolving a certain weight of the oil in an ethanolic KOH solution. The solution was then heated for half an hour in order to dissolve the oil completely in the ethanolic potassium hydroxide solution and the reflux condenser was attached to the flask. Then the mixture was brought to boil occasionally stirring using magnetic stirrer until the oil completely dissolved for half an hour. After the oil completely dissolved, 5 drops of phenolphthalein indicator was added and the hot soap solution obtained titrated with 0.5 M hydrochloric acid and the volume, V_a recorded). Similarly, a blank determination will be carried out on the same quantity of potassium hydroxide solution at the same time and under the same conditions and the volume, V_b recorded). The saponification value was thus calculated using the equation below:

$$\text{Saponification value} = \frac{56.1 \times M \times (V_b - V_a)}{W}$$

Where W is weight of oil taken in gram,

M is molarity of HCl solution,

V_a is volume of HCl solution used in the test in milliliter and

V_b is volume of HCl solution used in blank in milliliter.

Determination of Iodine Value

The method was described in AOAC [10]. A standard Wiji's solution was prepared by dissolving 9 g of iodine trichloride in 300 cm³ carbon tetrachloride and 700 cm³ of glacial acetic acid. The solution was left to stand for three days before use. Then *Jatropha curcas* oil was dissolved in 5 cm³ tetrachloride in a conical flask and 25 cm³ standardized Wiji's solution added and allowed to stand for 30 minutes at 20 °C in the dark. When the reaction was completed, 15 cm³ 10 % KI solution and 50 cm³ water were added. Finally, the free iodine was titrated with 0.1 M sodium thiosulphate until the color is pale yellow. A few drops of starch solution were added and the titration continued

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until the blue color was discharged. The volume of 0.1 M sodium thiosulphate was then recorded. Similarly, a blank determination was done for the same length of time and at the same temperature. The result was calculated using equation:

$$\text{Iodine Value} = \frac{1.269 (a - b)}{W}$$

Where W is the weight of oil used in the test
a is the cm³ of 0.1 M sodium thiosulphate required for blank determination and
b is the cm³ of 0.1 M sodium thiosulphate required for the titration.

Determination of Free Fatty Acid Value

The method described in [11]. First standard alcoholic KOH solution (0.1 M) was prepared by dissolving KOH (pellets) in ethanol. The solution was filtered and stored in an amber bottle for five days. Thereafter, a mixture of 95 % ethanol and diethyl ether in a ratio of 1:1 v/v was prepared by mixing 25 cm³ of diethyl ether and 25 cm³ of ethanol. A weighed quantity of the oil sample (2.5 g) was then dissolved in 25 cm³ of 1:1 mixture of ethanol and diethyl ether. The solution was then titrated with 0.1 M ethanolic KOH solution in the presence of 5 drops of phenolphthalein as indicator until the end point (colorless to pink) was recognized and the titrated volume recorded. The total acidity in mgKOH/g was then calculated using the following equation:

$$\text{Free Fatty Acid Value} = \frac{28.2 \times V \times N}{W}$$

Where V is the volume expressed in cm³ of 0.1 N solution of ethanolic KOH,
N is the normality of ethanolic KOH and
W is the weight in gram of the test portion.

Determination of Acid Value

The same procedure used for the determination of free fatty acid value was adopted for the determination of acid value except 0.1N NaOH was used instead of 0.1N of KOH [12]. The acid value was calculated using

$$\text{Acid Value} = \frac{56.1 \times V \times N}{W}$$

Determination of Specific Gravity

The method was described in [13]. *Jatropha curcas* oil (50 cm³) was measured into

a graduated cylinder and its temperature recorded. A hydrometer was then used to determine the density of the oil at 20 °C. The specific gravity was thus calculated according to ASTM D5355-95 as:

$$\text{Specific gravity (SG) of oil} = \frac{\rho_{oil}}{\rho_{water}}$$

Where ρ_{oil} is the density of oil in g/cm³
 ρ_{water} is the density of water at 20 °C in g/cm³

Determination of Moisture content

According to the method described in [14], about 4 g of the oil was weighed into a dish and dried in an oven at 105 °C for 7 hr, weighing every 2 hr till constant weight was obtained and the final weight taken was compared with the initially recorded weight. The percentage weight in the oil will be calculated using the formula:

$$\text{Moisture content} = \frac{W_2 - W_1}{W_1} \times 100$$

Where W_1 is the original weight of the sample before drying and
 W_2 is the weight of the sample after drying.

Production of Fatliquor from *Jatropha curcas* seed oil

The synthesis of the *Jatropha curcas* raw oil was carried in three (3) stages as described by Tawfig *et al.*, (2017):

i. Sulphation

163 g of *Jatropha curcas* oil was weighed into a conical flask. 20.38 g of 98% concentrated sulphuric acid (25 % of weight of oil) was put into the burette and was introduced to the oil in a drop wise manner. The conical flask was properly lined with ice block to maintain the temperature at 20 °C while stirring. This was continued until the entire acid was used up and then left for 24 hours.

ii. Washing

The resultant product above was then washed with brine solution (20 g of sodium chloride in 160 cm³ of distilled water) in a separating funnel. Then it was allowed to settle, the settle layer was then discarded. The remaining oil was transferred into a conical flask.

iii. Neutralization

The above product was then neutralized using 2 cm³ sodium hydroxide solution to pH of 7.

Physico-Mechanical Properties of Leather Fatliquored with Synthesized Fatliquor and Commercial Fatliquor

Tensile strength and percentage elongation were determined using test method of International Organization for Standardization ISO-3376: [16].

$$\text{tensile strength} = \frac{\text{breaking load (N)}}{\text{cross sectional area (mm}^2\text{)}}$$

$$\% \text{ elongation} = \frac{\text{final length} - \text{initial length}}{\text{initial length}} \times 100$$

Shrinkage temperature was determined using test method in accordance with ISO-3380 [17]. Resistance to Compression 'R' was calculated according to the Indian Standard Test Methods of Physical Testing of Leather [18] thus:

$$R = \frac{\text{increase in load per unit area}}{\text{decrease in thickness per unit thickness}}$$

$$R = \frac{(200 \times 4 / \pi) \times d_0}{1000 (d_0 - d_1)} \text{ kgf cm}^{-2}$$

Indentation index is measured as the difference between two mean thickness ($t_0 - t_1$) expressed in $1/100$ mm.

Thickness of samples were in accordance with ISO-2589 [19] using a standard measuring gauge. Abrasion resistance of leathers was carried out in accordance to ISO 17076-2 [20] using Martindale Abrasion Tester to test the abrasion resistance of the fabric while in constant friction with another material.

RESULTS AND DISCUSSION

Characterization of Raw *Jatropha curcas* Oil

The chemical analysis of the raw *Jatropha curcas* oil and synthesized fatliquor; were characterized and compared as shown on Table 1.

From Table 1, there was an observed increase in acid value after sulphation. This depicts the hydrolysis of the fatty molecules [21]. The free fatty acid value helps to determine the level of the oil's oxidative deterioration by enzymatic or chemical agents. From the results obtained, both values of 6.60 and 11.20 for raw *Jatropha curcas* oil (JO) and synthesized *Jatropha curcas* oil (SF) were low.

Table 1: Comparison between chemical properties of raw and synthesized fatliquor from *Jatropha curcas* oil

PROPERTIES	JO	SF
Acid value (mgNaOH/g)	13.20	22.40
Free fatty acid value (mgKOH/g)	6.60	11.20
Saponification value (mgKOH/g)	224.40	230.20
Iodine value (gl ₂ 100g ⁻¹)	72.12	60.00
	0.90	1.20
Specific gravity	8	20
Moisture content (%)		

This shows that the oil had not undergone considerable oxidative deterioration. This makes it suitable for use for further synthesis to fatliquor. The saponification values of 224.40 mgKOH/g and 230.20 mgKOH/g for raw *Jatropha curcas* oil and synthesized *Jatropha curcas* oil (SF) respectively were sufficiently high for use in fatliquoring. The high saponification value of oils depicts smaller molecules. This yields greater penetration power into the leather thereby increasing softness. The iodine values depict the degree of saturation or unsaturation of oils. The higher the

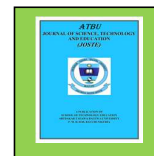
unsaturation of the oils, the more suitable they are for synthesis to fatliquor. This is because the sulphonic groups of the acid can sufficiently attack more unsaturated bonds, thereby enhancing better penetration. The iodine values of 72.12 for raw *Jatropha curcas* oil and 60.00 for synthesized *Jatropha curcas* oil (SF) respectively show a substantial degree of unsaturation. The moisture content of 8 % and 20 % for raw *Jatropha curcas* oil and synthesized *Jatropha curcas* oil (SF) respectively.

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There is an expected increase in the moisture content of the synthesized *Jatropha curcas* oil (SF) compared with the raw *Jatropha curcas* oil. This is imparted during the fatliquoring process.

Physico-Mechanical Analysis of Chrome Tanned Leather using Commercial Fat Liquor (CF) and Synthesized Fatliquor (SF)

Leather quality depends largely on the physico-mechanical properties of the finished leather. The physico-mechanical analysis of the leather tanned with chrome which was fatliquored separately with commercial and synthesized fatliquors, were tested and compared. The results are shown on Table 2.

Table 2: Comparison between values from physico-mechanical properties of fat-liquored chrome tanned leather from commercial liquor and synthesized liquor

PROPERTIES	CF	SF
Tensile strength (N)	24.41	30.00
Elongation (%)	21.00	60.00
Water Absorption (%)	18.31	18.33
Abrasion Resistance (%)	2.63	11.76
Shrinkage Temperature (°C)	96	96
Displacement (mm)	9.20	12.36
Resistance to Compression (kgf cm ⁻²)	2.62	2.88
Thickness (mm)	1.37	1.42
Indentation Index	23	23

From Table 2, an improvement in the physico-mechanical properties of the finished leather such as Tensile strength, Elongation at break, Abrasion Resistance, Thickness and Displacement (Lastometer test) were observed. This was similar to the work by El-Shattory, *et al.*, [22] where the ethoxylation process was employed to produce a non-ionic surfactant from JC oil using K₂CO₃ as a catalyst. The fatliquored leather obtained from fatty ethoxylate produced was observed to improve mechanical properties such as tensile strength and elongation at break. Tensile strength is the value of load a sample's cross-section can bear without breaking when loaded in the axial direction, and it is related to the leather sample's strength of the collagen fiber bundles. The tensile strength can also reveal the flexibility of its collagen fibres which is an important property of fatliquored leather and its structural resistance [23]. The tensile strength values of the leathers fatliquored both with the SF and CF were above the recommended standard value of 15 N/mm² for shoe upper leather. Although, the SF gave a higher value for tensile strength than that of the CF which makes it more tear resistant. This can be attributed to the chemical

properties of the fatliquor used. The elongation at break can characterize the softness, flexibility, strength and toughness of a leather sample. It should not be excessive to avoid the ease of footwear deformation [23]. The recommended standard value for percentage elongation is within 40 % to 70 %. For the results obtained, the SF gave value of 60 % as against CF whose value of 21 % was below desirable. This can be attributed to the nature of the original *Jatropha curcas* oil used. The lastometer test revealed a higher value of displacement for the SF (12.36 mm) as against 9.2 mm for CF, which both falls above the recommended standard of 7 mm. This means that the leathers fatliquored by both SF and CF can be used to produce shoe upper leathers since it will be easily shaped.

The water absorption property of natural leathers shows its ability to absorb and transmit moisture (breathe), also its ability to insulate in cold weather conditions and cool in hot weather conditions [24]. The water absorption of 18.31 % for CF and 18.33 % for SF were both suitable for leathers. Chrome-tanned leathers could reach shrinkage temperatures above 100°C. The shrinkage

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temperature of 96°C and indentation index of 23 for both CF and SF are in accordance with the work by Akawu [25] showing the hydrothermal stability of the fatliquored leathers. The abrasion resistance is the property related to the resistance a material can handle when subjected to friction. From the results obtained, values of 2.63 % and 11.76 % for CF and SF were obtained. Thus the SF will produce leathers with better resistance to friction, which is desirable. The resistance to compression is significant in providing information on the surface characteristic and shape retention properties for shoe upper leather. From the results obtained, values of 2.62 kgf cm⁻² and 2.88 kgf cm⁻² for CF and SF respectively. The higher the resistance to compression, the fuller the leather. Full leather is desired because of its high economic value which makes SF more preferred here. The thickness is also a measure of the fullness of the leather. The SF gave a thicker thus fuller result of 1.42 mm as against the CF of 1.37 mm, making it better desired.

CONCLUSION

In conclusion, vegetable oils have been known to be sustainable as a raw material for synthesizing fatliquors. From the various analyses carried out on the raw and sulphated *Jatropha curcas* oil, the synthesized fatliquor could serve as a good alternative for imported fat liquors as it meets the required leather standards. The leathers will also be suitable for use as shoe upper leather.

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