Evaluation of the leather fatliquoring potential of sulphonated *Afzelia africana* aril cap oil

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**ABSTRACT**

The synthesis, properties, characterization and sulphonation of *Afzelia africana* aril cap oil were examined to establish its leather fatliquoring potential. The analysis of the *Afzelia africana* aril cap oil before and after the completion of sulphonation process was carried out in order to confirm the modification of the oil. The physicochemical properties showed a marked difference between both analyzed oils as the melting point, acid value, free fatty acid, iodine value, saponification value and % SO3 of the unsulphonated/sulphonated oil were observed to be 6.39 °C; 19.90 °C, 12.99 mg KOH/g; 0.50 mg KOH/g, 6.50; 5.25, 77 g iodine/100g; 21 g iodine/100g, 185 mg KOH; 176 mg KOH and nil; 3.92 % respectively. The fatliquoring potential was examined and compared with commercial fatliquor using standard methods. A significant improvement in the lubrication and mechanical properties of the leather treated with the sulphonated *Afzelia africana* aril cap oil was revealed by Sudan stain and Tensile strength; Double edge Tear; Elongation at break test results respectively. The improved lubrication and mechanical properties of the treated leather compared favourably with commercial fatliquor. This study shows that aril caps of *A. africana* of no commercial value can be a source of fatliquor for the leather industry.

1. Introduction

The extraction and use of vegetable oils has for centuries played an important role in the manufacture of a large number of industrial products and food items. Such industrial applications include the use as lubricants in the leather industry. The process of converting hides or skins into leathers by sequential mechanical and chemical steps is known as leather tanning [1]. Tanning is very important in leather processing. The process of converting hides or skins into leathers and food items. Such industrial applications include the use as lubricants in the leather industry. The process of converting hides or skins into leathers by sequential mechanical and chemical steps is known as leather tanning [1]. Tanning is very important in leather processing.

*Afzelia africana*, also known as the African oak, is a deciduous plant widely distributed in many regions of Africa. It belongs to a family known as Fabaceae, sub-family Caesalpiniaceae and can usually be found in humid and dry forests [6]. This plant can grow into a large tree up to 25–30m in height, has multiple pods which bears 6–10 hard shining black oblong shaped seeds with sweet bright orange aril cap in one third of its length from the base [6, 7]. In Nigeria, the flour obtained from the seeds is used as soup thickeners, while the aril caps are often discarded and have no use [6]. Neither do they have any market value.

Fatliquors are usually imported from other countries to Nigeria and as such the invested enormous, foreign currency on this non-domestic commodity is outrageous. This study therefore attempts to prepare a substitute of imported fatliquors from our local raw materials. *Afzelia africana* aril cap oil. The oil was sulphonated and its potential application introducing softness and safeguarding the leather against cracking [2, 3]. Physical characteristics of leather such as flexibility, feel, and stitch tear resistance are usually influenced by the nature of fatliquors used. The suitability of a particular oil as a major raw material in growing commercial fatliquor industry is determined by its abundance, existing usage, odour and colour [5].

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in the leather industry as a leather lubricant/fatliquor in shoe upper manufacture was assessed.

2. Materials and methods

Matured dry pods of (A. africana) were obtained from a farm in Ndufu-Alike town, Ikwo, Ebonyi State, Nigeria. Samples of the seeds were authenticated at the Herbarium Unit of the Department of Biological Sciences, Ahmadu Bello University, Zaria, Nigeria. The pods were cut open and the bright-orange coloured aril caps were carefully detached from the seeds. These aril caps were dried in an oven at 40 °C for 5 h, crushed (approximately 2 mm) and the oil was extracted with a soxhlet extractor using n-hexane as the solvent. Figure 1 shows images of Afzelia africana aril caps, Afzelia africana aril caps seeds embedded in pods and Afzelia africana aril caps seeds.

Wet blue goat skins were obtained from the tannery at the Institute for Creative Leather Technologies (ICLT), The University of Northampton (UoN), Northampton, United Kingdom. Reagents used in the laboratory for synthesis and analysis were of analytical grade while those used for leather processing were of commercial/industrial grade.

2.1. Physicochemical properties determination

Physicochemical properties of (A. africana) aril cap oil like acid value (Cd 3a-63), specific gravity (Ta 1b-64), saponification value (Cd 3–25), iodine value (Cd 1–25) were determined by American oil chemists' so-ciety (AOCS) methods [8].

2.2. Determination of fatty acid composition

The fatty acid composition of A. africana aril cap oil (ACO) was determined using Gas Chromatography-Mass Spectroscopy, GC-MS. This was done using its methyl ester prepared with the method described by Adewuyi et al. [9] on an Agilent19091S–433HP-5MS gas chromatograph attached to a mass spectrometer. The injection and detection temperatures were 280 and 300 °C respectively. The carrier gas used was Helium at a flow rate of 20 ml/min. The area percentages were recorded with a standard Chemstation Data system. For the mass spectrometry, an ACQ mode scanner (with scan range of 15–500 amu and voltage of 2094) was used and the mass spectra were compared with the NIST11 mass spectral library.

2.3. Sulphonation process

The sulphonation of Afzelia africana aril cap oil was carried out as described by Nkwor et al. [10]. In a typical experiment, concentrated sulphuric acid (45 ml) was added dropwise into 150 g of (A. africana) aril cap oil (with constant stirring at 20 °C for 2 h). The crude mass was dissolved in 450 ml of ethanol, and neutralised using 15 % NaOH (solubilised in methanol) (Schemes 1 and 2). The salts were filtered off under vacuum and solvent recovered via a rotary evaporator. The resulting sulphonated product was ready for use as a leather fatliquor.

The side reaction can be found below:

2.4. Melting point determination

Differential scanning calorimetry (DSC) of the oil was performed using a DSC 2 Star System (Mettler Toledo). The purge gas (nitrogen) had a flow rate ~60 ml/min 5–7mg of oil were weighed into low pressure aluminium crucibles, and sealed hermetically. The sealed crucibles were pierced prior to analysis [11]. An empty, hermetically sealed aluminium crucible with a pinhole was used as a reference. A temperature profile of -80 to 180 °C was run using the following temperature program: -80 °C isotherm for 3 min; dynamic ramp at -80 °C–180 °C (at 10 °C min−1), isotherm at 180 °C for 3 min; isotherm at 30 °C for 2 min. The resulting

Figure 1. Images of Afzelia africana: a – aril caps, b – seeds embedded in pods, c – seeds.

Scheme 1. Sulphonation of A. africana aril cap oil to produce sulphonated (Sulphated) oil.

Scheme 2. Side by side reaction of the sulphonation of A. africana aril cap oil.
DSC data was analysed for peak temperature, onset temperature and melting temperature for comparison. All experiments were carried out in triplicate and the average values were reported. Melting temperature was considered to be the temperature at the end of the melting transition [12].

2.5. Characterisation of the sulphonated oil

The presence of H–C–S and H–C–O–S groups in the sulphonated *Afzelia africana* aril cap oil (fatliquor) were examined and characterised by FT-IR measurement (600-4000 cm⁻¹), normal resolution of 4 cm⁻¹ using a Shimadzu 8400S FT-IR instrument (Shimadzu, Milton Keynes, UK). ¹H nuclear magnetic resonance (NMR), ¹³C NMR and distortionless enhancement were determined by polarization transfer (DEPT) ¹³C NMR. The spectra of both the sulphonated and unsulphonated oils were acquired on a Bruker Biospin AV500 – 5mm BBO probe with Z axis gradient, TOPSPIN v 2.1, 1H = 500.13 MHz, 13C = 125.76 MHz (Bruker, Coventry, UK). Their thermal behaviours were also determined using the Mettler DSC 2 Star System in temperature range of -80 to 180 °C, using an identical program given.

2.6. Physicochemical properties determination of the sulphonated oil

The physicochemical properties were determined according to the standard methods recommended by the Society of Leather Chemists and Technologists [13].

2.7. Fatliquoring process

Fatliquoring process was carried out as described by Institute for Creative Leather Technologies leather shoe upper manufacture manual [14]. Selected wet blue goat skin (without defects such as scratches and flap cuts) was shaved to obtain a uniform thickness (1.2–1.3 mm) in the butt area. The butt was divided into four quarters such that the sampling positions (BS EN ISO 2418) [15], were uniformly represented in all the four quarters. Three of the quarters, labelled NC (Negative control (without fatliquor)), PC (Positive control-with reference commercial fatliquor- Trupon DXV (Trumpler Gmbh, Worms, Germany), a common imported sulphated fatliquor used in the processing of leather) and Sulphonated *Afzelia africana* Aril Cap Oil were used for proper comparison. Treatments on the three quarters were simultaneously carried out (with the aid of three separate tanning drums) using a conventional shoe upper manufacturing process (fatliquoring process) [14]. The chrome tanned goat skin (400 g) was wet back by the addition of water (300 %) and wetting agent-Bermanol WAU (0.2 %) in the drums at a temperature of 30 °C and sodium bicarbonate (0.25 %) for 30 min at 35 °C, neutralised by addition of water (100 %), sodium formate (1%) for 5 min and sodium bicarbonate (0.25 %) for 30 min at 35 °C, drained, washed with water (200 %) and drained again. On addition of water (100 %) and replacement syntan (Trupotan GDL) (6 %) in the drum, it was run for 15 min and the vegetable tannin was added at 30 °C and allowed to run for another 30 min. Water (200 %) and Acrylic resin (3 %) were added and allowed to run for another 30 min at 35 °C before drainage. It was further washed with water (200 %) for 5 min at 50 °C and drained. Sulphonated *Afzelia africana* aril cap oil, mixed with water (1:3) (8 %) was added and allowed to run for 50 min at 50 °C (Note that the Commercial fatliquor was added in the second drum in place of SACO while no fatliquor was put in the third drum as a negative control). Formic acid (1 %) was added and allowed to run for 20 min, washed for 10 min twice and horse dried. (Also note that dyeing, which is usually carried out before the addition of fatliquor was omitted in the leather processing to make the analysis of the extent of penetration of the oil (sudan stain test) possible).

2.8. Mechanical properties of leather

All leather samples: NC, PC, SACO were conditioned according to BS EN ISO 2419 [16] prior to staking twice using a Cartigliano PAL 160 leather staking machine (Cartigliano) and subsequent mechanical testing.

The mechanical properties of leather samples were all determined using softness (BS EN ISO 17235) [17], tensile strength (BS EN ISO 3376) [18], elongation at break and tear strength of leather (BS EN ISO 3377-2) [19] and grain strength standards (BS EN ISO 3379) [20].

Thin sections (50 μm) of the leather samples were cut with a Leica 1850 cryostat microtome (Leica, Wetzler, Germany) (set at -20 °C) and used in the Sudan (IV) stain test for the determination of extent of penetration of the fatliquors into the leather fibrils.

3. Results and discussions

3.1. Physicochemical properties

Table 1 shows the Physicochemical properties of the Unsulphonated and Sulphonated *Afzelia africana* aril cap oil. The high percentage yield of the *Afzelia africana* aril cap oil revealed by the results (Table 1) indicates its availability and sustainability for commercial use. The moderate value of iodine shows a reasonable amount of unsaturation that is readily available for sulphonation reaction [21]. The sulphonated oil synthesized was virtually free from inorganic salts. The percentage SO₃ of 3.92 % observed for the sulphonated oil is an indication that some degree of sulphate group has been incorporated into the *Afzelia africana* aril cap oil (Table 1).

One of the factors widely used in determining the suitability of a surfactant for a given application is its Hydrophile–Lipophile balance (HLB) result [22]. HLB is a measure of a surfactant partitioning tendency between oil and water. Calculating the Hydrophile/Lipophile Balance (HLB) of a fatliquor is usually a difficult task because the precise structures of fatliquors are usually unknown [5, 23]. However, a method of determining HLB via estimations made by carrying out observations on 10 % emulsions of the fatliquors was established by Waite [23]. It was established that the lower HLB materials will always give a milky colloidal emulsion, while higher HLB values usually gives clear colloidal solutions [23].

It could therefore be suggested that since the 10 % emulsion used in this study gave a translucent solution, the HLB value may be relatively high. This therefore predicts that the emulsion is an oil in water emulsion.

Table 1. Physicochemical properties of the *Afzelia africana* aril cap oil.

| Parameter                      | Unsulphonated oil | Sulphonated oil |
|--------------------------------|-------------------|-----------------|
| Colour                         | Dark Orange       | Brown red       |
| Percentage yield (%)           | 55.39             | 68.8            |
| Specific gravity (g/cm³) (at 20 °C) | 0.941             | 0.956           |
| pH of 10 % emulsion            | Not Applicable    | 7.54            |
| Stability of 10 % solution     | Not Applicable    | Stable >24hrs   |
| % Ash                          | -                 | Trace           |
| Appearance of 10% solution     | Not Applicable    | Translucent    |
| Colour of 10% solution         | Not Applicable    | Pale Orange red|
| Melting point (°C)             | 6.39              | 16.87           |
| Acid Value (mg KOH/g)          | 12.99             | 10.50           |
| Free fatty acid (as oleic acid)| 6.50              | 5.25            |
| Iodine Value (g iodine/100g)   | 77                | 21              |
| Saponification value (mg KOH/g)| 185               | 176             |
| % SO₃                          | Not Applicable    | 3.92            |
3.2. Fatty acid composition

Table 2 shows the types of fatty acids and the percentage composition profile of *Afzelia africana* aril cap oil. A greater proportion of unsaturated fatty acids (58.94 %) was found in the oil and this indicates that there are many double bonds that could readily be available for the sulphonation reaction.

| Fatty acid                | Percentage composition |
|--------------------------|------------------------|
| Palmitic acid (C16)      | 33.84                  |
| Stearic acid (C18)       | 7.22                   |
| Saturated fatty acids    | 41.06                  |
| Oleic acid (C18:1)       | 30.04                  |
| Linoleic acid (C18:2)    | 28.90                  |
| Unsaturated fatty acids  | 58.94                  |

3.3. DSC analysis

These melting ranges and DSC curves (Figure 2) result from combined effects of the fatty acid composition, polymorphism of natural oils and fats and thermal history [24, 25]. These results have been summarised in Table 3. According to Berg et al. [26], unsaturated fatty acids have lower melting points than saturated fatty acids. The increase in the melting point found in the sulphonated oil (Table 3) shows that most of the unsaturation in the fatty acids content of the oil have been used up during the sulphonation reaction; leaving behind fatty acids with high degree of saturation (which have a higher melting point) than unsaturated fatty acid.

![Figure 2. DSC result of (a) Unsulphonated Aril Cap Oil (ACO) (b) Sulphonated Aril Cap Oil (SACO).](image-url)
3.4. FT-IR analysis result

Figure 3(a & b) show the functional groups present in the unsulphonated and sulphonated *Afzelia africana* aril cap oil with different level of intensities. The peak at 3009 cm\(^{-1}\) can be assigned to C–H stretching frequency of non-conjugated unsaturation (Figure 3 a), whereas, there was no peak at this wave number in Figure 3b. This may be due to the attack of H\(_2\)SO\(_4\) on the –C=C- bond in the oil sample to form –C=C- sulphonated oil product. The peak at 3464 cm\(^{-1}\) can be credited to the traces of alcohol used in the formation of the sulphonated product (Figure 3b). The emergence of peak at 1200 cm\(^{-1}\) in Figure 3b can be assigned to S=O stretching of both sulphate and sulphonate groups. The absence of this peak (1200 cm\(^{-1}\)) in Figure 3a further confirms the incorporation of sulphonate group in the *Afzelia africana* aril cap oil. The peaks at 2853 cm\(^{-1}\) can be assigned to C–H stretching frequency of alkane (Figure 3a, b). The peak at 1744 cm\(^{-1}\) can be attributed to the presence of C=O stretching frequency of ester (Figure 3a, b). The peak at 1464 cm\(^{-1}\) can be ascribed to the bending frequency of unsaturated alkene (Figure 3a, b). The observed peak at 721 cm\(^{-1}\) can be attributed to the bending frequency of saturated oil. Table 4 shows the main FT-IR

| Oil Sample | Onset Temperature (°C) | Peak Temperature (°C) | Endset Temperature (Melting Point) (°C) |
|------------|------------------------|------------------------|----------------------------------------|
| ACO        | -15.60                 | -78.47 × 10\(^{-3}\)   | 6.39                                   |
| SACO       | -3.73                  | 11.30                  | 16.87                                  |

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peaks of the unsulphonated and sulphonated Afzelia africana aril cap oils and their corresponding functional groups.

3.5. NMR analysis result

Figure 4 depicts the $^1$H NMR spectra of the studied Afzelia africana aril cap oil before and after sulphonation. The spectra show about nine to ten signals of significant intensity for both oil samples. The terminal methyl group gave a δ of about 0.85 ppm. The chemical shift between 1.1 ppm and 2.2 ppm shows the methylene proton signals at various positions of the acyl chain as assigned in the triglycerol structure. Peaks at δ 4.11–4.32 ppm are attributed to protons of glyceride moiety, while the peak at δ 5.26–5.35 ppm are assigned to the protons of the –CH = CH– moiety. Similar assignments have been reported by several studies [27, 28]. These protons of the –CH = CH– moiety are sp$^2$ hybridized and as such their NMR signals are deshielded by the influence of the diamagnetic anisotropy of the π system. Sulphation or sulphonation usually leads to the saturation of the double bond. The sp$^3$ hybridized protons formed are thus expected to be shielded relative to the sp$^2$ olefinic protons. The newly formed protons (H–C–S or H–C–O) in the sulphonated oil showed signals at δ 3.65 and 3.73 ppm.

It is important to note that the slight deshielding observed for these protons relative to the rest of the protons in the sulphonated oil may have been due to the electron withdrawing effect of sulphur and oxygen atoms. The inductive effect, however, causes less deshielding than diamagnetic anisotropy. These proton signals at δ 3.65 and 3.73 ppm are completely absent in the unsulphonated oils. The proton δ at 2.72 ppm due to CH–CH–CH2–CH=CH were almost absent in the sulphonated oil.

Figure 5 shows the $^{13}$C NMR spectra of the unsulphonated and sulphonated Afzelia africana Aril Cap Oil. The methyl groups at the end of the acyl chains in glyceride moiety for the unsulphonated Afzelia africana Aril Cap Oil gave a signal at around 14.1 ppm (Figure 5a). It is well separated from other signals and hence easily recognizable. The signals associated with the olefinic carbons was observed to be highly deshielded at δ 127–131 ppm due to the diamagnetic anisotropic effect of the π system. Upon sulphonation, these signals disappeared completely due to loss of the double bonds (Figure 5b). The new signals which appeared at 52 and 72 ppm can be ascribed to the sp3 hybridized carbons (C–S and C–O) formed after the sulphation or sulphonation reactions. The slightly deshielded position of these signals observed in the spectra is also due to the influence of the electron withdrawing effect of sulphur and oxygen.

| Table 4. The main IR peaks of ACO and SACO and their corresponding functional groups. |
|---------------------------------------------------------------|
| **Frequency cm$^{-1}$** | **Assignment** | **Remark** |
|--------------------------|-----------------|-------------|
| Unsulphonated Oil (ACO) | Sulphonated Oil (SACO) |  |
| -3464 | O–H | The traces of alcohol used in the formation of the sulphonated product |
| 3009 | C–H | Stretching frequency of non-conjugated unsaturation |
| 2853 | C–H | Stretching frequency of alkane |
| 1745 | C–O | Stretching frequency of Ester |
| 1199 | S–O | Stretching of both sulphate and sulphonate groups. |
| 1464 | C–H | Bending frequency of unsaturated alkene |
| 721 | C–C | Bending frequency of saturated carbon atom |

Figure 4. $^1$H NMR Spectra of (a) unsulphonated Afzelia africana aril cap oil (b) Sulphonated Afzelia africana aril cap oil.
atoms (Figure 5b). This was also in agreement with the iodine value of the sulphonated *Afzelia africana* aril cap oil which decreased significantly when compared with that of the unsulphonated *Afzelia africana* aril cap oil.

The $^{13}$C NMR DEPT spectra of the unsulphonated and sulphonated *Afzelia africana* Aril Cap Oil is displayed by Figure 6. The terminal CH$_3$ was observed to be in the opposite direction compared to other peaks in both spectrum at $\delta$ 14 ppm (Figure 4(a,b)). In like manner, the C–H–O of the glycerol backbone can be attributed to $\delta$ 68 ppm. The two CH$_2$O of the glycerol backbone were assigned to $\delta$ 64 ppm and 62 ppm. The -(CH$_2$)$_2$ of the fatty acid chains were linked to various positions at $\delta$ 20–30 ppm. The evidence of the formation of C–O–S and C–S bonds by the reaction with H$_2$SO$_4$ was confirmed by the absence of the HC = CH bond previously found at $\delta$ 127 ppm and 131.87 ppm in unsulphonated oil, ACO. The C–O–S was ascribed to $\delta$ 65.27 ppm in the sulphonated oil, SACO and C–S bonds were equally assigned to various positions at $\delta$ 60.16 ppm and 58.38 ppm. These C–O–S and C–S bonds were completely absent in the unsulphonated oil.

### 3.6. Staining test result

Figure 7 shows the staining test carried out on Chrome Tanned Goatskin in the absence of fatliquor, the presence of sulphonated *Afzelia africana* Aril Cap Oil (Fatliquor) and presence of commercial sulphated fatliquor. The efficiency of a fatliquor for leather depends on how deeply it is able to penetrate into the hierarchy of the fibre bundle. The red
colour stain seen in Figure 7b and 7c is an indication that there was a deep penetration of the sulphonated *Afzelia africana* Aril Cap Oil (fatliquor) and the Commercial sulphated fatliquor. The red colour seen in both images (Figure 7a, b) indicates the presence of fats. The absence of red stain in the image displayed in Figure 7a shows that there is no fat present in the chrome tanned goatskin cross-section (a).

3.7. Mechanical properties of fixed leather samples

3.7.1. Softness test

Table 5 illustrates the softness property of the cross sections of the leather. The softness test results of both the SACO and PC are within the same range (comparable) unlike the result from the negative control, NC. The softness result therefore conforms to the result obtained from the Sudan stain test.

3.7.2. Strength properties

Table 6 shows the strength properties of the leather without Fatliquor (NC), leather with commercial Sulphated Fatliquor (PC) and leather with the Sulphonated *Afzelia africana* Aril Cap Oil (Fatliquor) (SACO). The negative control (i.e. leather without fatliquor) was observed to possess the lowest values of tensile strength and elongation at break (having its mean tensile strength value as 17.66 N/mm² and average elongation at break as 27.52 %) when compared to the leather with commercial Sulphated fatliquor (PC) and leather with Sulphonated *Afzelia africana* Aril Cap Oil (Fatliquor). The PC and SACO had higher tensile strength values of 24.97 and 19.56 N/mm² respectively and average elongation at break as 38.56 % and 40.42 % respectively.

The results show that sulphonated *Afzelia africana* aril cap oil (fatliquor) is evidently comparable with Trupon DXV, a commercial sulphated fatliquor with respect to its strength properties.

4. Conclusion

This study evaluates the leather fatliquoring potential of sulphonated *Afzelia africana* Aril Cap oil. The physico-chemical, mechanical and performance characteristics of the as–synthesized sulphonated
Afzelia africana Aril Cap oil compared favourably with commercial leather fatliquor. The results showed that Afzelia africana Aril Cap oil of no commercial value can be a source of fatliquor for the leather industry.

### Declarations

**Author contribution statement**

Adachukwu Nkwor: Conceived and designed the experiments; Performed the experiments; Analyzed and interpreted the data; Contributed reagents, materials, analysis tools or data; Wrote the paper.

Pius Ukoha: Analyzed and interpreted the data; Contributed reagents, materials, analysis tools or data.

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**Competing interest statement**

The authors declare no conflict of interest.

**Additional information**

No additional information is available for this paper.

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