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Synthesis Of Sulphated-Fatliquor From Neem (Azadirachta Indica) Seed Oil For Leather Tannage.

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ABSTRACT:*Fatliquor affects the physical properties of the leather and makes it more flexible and softer. Fatliquorprepared from Neem seed oil by sulphation process using sulphuric acid followed by addition of sodium hydroxide to maintain pH at 5.0 with conc. FT-IR analysis of the sulphated product confirmed the attachment of sulphonicacid group. The physical and chemical properties of the fatliquor were found satisfactory. The preparedfatliquor was used for the processing of goat skins. The processed goat skins physical and chemicalproperties compared with the skins processed by imported palm oil fatliquor. Goat skins processed by these two types of fatliquor observed standard leather specification. So fatliqour prepared from Neemseed oil extracted by soxlet extractionmethod may be considered as a substitute for imported fatliqour.*
KEYWORDS*: Fatliquor, FT-IR, Strength properties, organoleptic properties, sulphation, Neem seed oil.*

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I. INTRODUCTION

The transformation of hides and skin into leather requires several chemical and mechanical operations; such as the Pre-and post tanning operations. The pre-tanning operation (beam house operation), such as soaking, fleshing, unhairing/liming, division, deliming, bating and pickling, are made to clean the hide and skin and remove non-protein matter(Burgess, 1994). While, the fleshing operation removes flesh and natural fats from the skins. The presence of fats accelerate skin degradation and fats do not form the leather structure(Covington,2009). Besides, fat layer makes the penetration of tanning chemical through the the leather structure difficult. Thus the fat removal improves tanning efficiency and prevent formation of undesirable products, such as soaps (Roaldi, *et al*, 2010).However, the removal of fat makes the leather when dried stiff, and brittle (Thorstensen, 1993).Stiff leather according to Kronick (1996) cannot be worked. The preliminary beam house processes remove most of the natural oils from the hides and skins after tannage.

The requisite physical characteristics such as feel, softness, water repellency, tear resistance, break, and tensile strength, as well as comfort properties of leathers (Covington, 2001) can only be attained by introducing fatliquor into the leather. (Thorstensen,1993).

Technically, fatliquors are surface-active softening agents used as lubricants in leather(Zhang, *et al*, 2004). It is produced by emulsification process of introducing sulphate or sulphite groups into the structure of oils and fat (Reich, 2007). According to Covington (1993), the traditional raw materials used for fatliquor production has for many decades been fish, animal, edible vegetable oil and mineral oil. Due to the presence of high amount of long, unsaturated carbon chains, fish and vegetable oil fatliquor gives superior softness to the leather. However,with the increasing price of edible oil and the need to produce eco-friendly leather articles, it no longer sustainable to use fish and edible vegetable oil thus non-edible oil such as Neem seed oil may be a better substitude.

Neem oil is a non-edible vegetable oil obtained from the seeds of the Neem plant (Azadirachtaindica). It is golden yellow or dark brown, in colour. It has a strong, pungent odour, and biter taste.It is composed mainly of triglycerides and triterpenoid.It is used worldwide as medicine(Marcello, *et al*.,2012).

In this study, attempt has been made to evaluate the potential of using fatliqour prepared from Neem seed OIL infatliquoring light leathers.The use of neem oil to synthesizefatliquor as an import substitute ensures a significant cut in import cost. The effectiveness of the prepared fatliquoron light leathers is also compared against the imported palm oil fatliquor.

II. EXPERIMENTAL PROCEDURE

Collection and processing of seeds

Neem seeds were purchased from Central market, Maiduguri, Maiduguri Local Government Area of Borno State, Nigeria. The fleshy part of the fruits was removed by washing in clean water and then sun-dry in an open air for five days. The seeds were cracked manually to remove the shells and hulls. The kernels was ground into powder using pestle and mortar. The powder called neem kernel meal (NKM) was used for oil extraction.

Extraction of Neem Oil (Sani, 2013)

100g of neem seeds powder was mixed with 200 ml n-hexane (boiling point 40–60^{o}C)insoxhlet extraction kit and heated at 60^{o}C for six hours. The oil extracted wasrefluxed at 70ºC for three hours to remove the solvent usedand was centrifuged at 5000 rpm for 20 minutes to remove suspended particles.The process of extraction was repeated until sufficient quantity of the oil was obtained.

Yield of oil (%) = Mass of oil/ initial mass of sample× 100

Physico-chemical properties of neem oil extracted

Determination of Moisture Content

First the empty dish was weighed. then 4 g of neem powder into an empty dish. The sample was dried in an oven at 105^{0}C for 6 h, and was weighed every 2 h till constant weight was obtained The percentage of moisture weight was calculated using the formula:

Moisture content =

(w1 - w2)/w1 * 100

w1 is original weight of the sample before drying and

w2 is weight of the sample after drying.

Determination of Specific Gravity.

Specific gravity Hydrometer was used to measure the specific gravity of the oil at 25^{o}C specified

DETERMINATION OF ACID VALUE(AOCS, 2015)

To determine the Acid value, a mixture of 95% ethanol and diethyl ether in a ratio of 1:1 v/v was prepared. 2 g of the oil sample was dissolve in 25 ml ethanol-diethyl ether mixture prepared and was heated to boil. The solution obtained was titrated against 0.1N KOH solution using 5 drops of phenolphthalein as indicator until a pink colouration was obtained. The volume of KOH used wasnoted. The total acidity in mg KOH/gm was calculated using the equation:

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

Where; V = the volume of KOH,

N = concentration KOH and

W= weight in gram of the sample

DETERMINATION OF IODINE VALUE

To determine the iodine number, 3g of oil was dissolved in 5ml carbon tetra chloride CCl_4 (chloroform) in a glass stoppered conical flask and 25 ml Wij's solution was added and the flask was allowed to stand for 30 minutes at 20^{0}C in a closed cupboard. When the reaction is completed, 15 ml 10% KI solution and 50 ml distilled water was added. Finally the free iodine liberated was titrated with 0.1N sodium thiosulphate using two drops of starch solution asindicator, until the blue colour formed disappeared. The volume of 0.1N thiosulphate will be recorded. Similarly a under the same condition a blank determination was set up.The result was calculated using equation:

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

Where; W = the weight of oil used in the test,

a = volume of 0.1N thiosulphate used for the blank determination and

b = volume of 0.1N thiosulphate used for the titration.

III. DETERMINATION OF SAPONIFICATION VALUE OF NEEM SEED OIL

2 g of oil was added to 25 ml of 0.5N ethanolic potassium hydroxide solution and the reflux condenser was attached to the flask. Then the mixture was heated, and as soon as the ethanol boils, the flask was occasionally shaken using magnetic stirrer until the oil was completely dissolved, and the solution was boiled for half an hour. After the oil was completely dissolved, 5 drops of phenolphthalein indicator was added and the

hot oil solution obtained was slowly titrated with 0.5N hydrochloric acid (and volume Va was recorded). Similarly a blank determination was carried out upon the same quantity of potassium hydroxide solution at the same time and under the same conditions (and volume Vbwas recorded). The final result was calculated using equation:

Saponification value $= \frac{56.1 \times M \times (vb - va}{W}$

Where W = weight of oil (g),
M = concentration of HCL ,
Va = volume of HCL used in the test
Vb = volume of HCL used in blank

Synthesis of neem oil fatliquor by Sulphation(Ariful, et. al,2015)

40 ml of concentrated sulphuric acid was added drop-wise from a clamped burrette into 200g of need oil with constant stirring using mechanical stirrer at 20°C temperature.The reaction was carried outslowly for 3 hours. The sulphated product was shaken with 400 ml of10% sodium chloride solution and then kept in a separating funnel overnight. The bottom layer was discarded to obtain fatliquors. The pHof thefatliquor was adjusted to pH 5.0 by adding 20ml, 30% sodium hydroxidesolution and the resulting fatliquor was bottled and stored at room temperature

Physico-chemical Characterization of Neemoil Fatliquor(BIS. 2008)

Stability of Emulsion Tanning salt solution

10%fatliquor emulsions were prepared by dispersing 10ml of fatliqour in 90 mL of distilled water. The pH of the emulsion and percentage phase separations at 25°C was determined.20ml of the 10% Fatliquor emulsion was mixed with 5% solutions of NaCl, MgSO4 and basic CrSO4in separate 100ml measuring cylinder and allowed to stand for a night. The percentage phase separation as a function of time was determined.

Determination of Total Active Ingredient, IS

5 g of the neem oil fatliquor was weighed into a 250 ml flask and 25 ml of50% ethanol and 25 ml of petroleum ether was added.Then the contents was transferredinto a separating funnel and shaken vigorously. It was mounted on a restord stand for a night. Thelower alcohol layer was transferred to another separating funnel and it was extracted fourtimes with petroleum ether. The upper layer the petroleum ether layer, was extracted, with 75% alcohol and the layers was allowed to separate and further extracted with 90%alcohol and absolute alcohol; and the layers was allowed to separate. The petroleum etherlayer and alcohol layer were collected into two separate flasks. The solvents was evaporated, and theresidues dried to constant weight in an oven; cooled and weighed. Alcohol layer contains thefatliquor and the petroleum ether layer contains the neutral oil. The calculation was doneusing equations given below.

% Free oil, (A) = F/ W× 100
% Emulsifier(SO_3) (B) = E/ W× 100
WhereF =weight of petroleum ether soluble(g)
E =weight of alcohol soluble (g) and
W =weight of the fatliquor sample(g).
Total active ingredient, in percent = A + B

Application of Neem Oil and Commercial fatliquors separately on goat skin leather (Alexander, *et al.*, 2015).

Full chromed goat wet-blue leather was cut intotwo equal parts by mass; one part was fatliquoured with neem oil fatliquor, while the orderpart wasfatliquored with palm oil commercial fatliquor (control).Two tanning drums were used for this experiment.

Characteristerizaion of Fatliquored Leathers

Organoleptic Tests

Hand Evaluation (INESCOP,2013)

The fatliquoredsamples and control leather were secretly coded A and B for hand and visual examination of fullness, softness, grain tightness (break),odor, colour andsurface touch by fiveexperienced leather technologists. The grading was rated on a scale of 1-5 points for each functional property. A value of one was for the lowest and 5 for the highest property.

Physical resistance tests

Tensile strength and adhesion of finish(INESCOP,2013)

The tensile strength was determined using a tensile tester. Test pieces was cut from the leather. One centimeter strip of polythene was sticked to one end of the specimen using super glue. The specimen was subjected to finish tester to determine the strip off weight. The tensile strength was calculated using equation given below.

Tensile strength(N/mm^2) = breaking load / thickness (mm) × width (mm)

Flexing Endurance(INESCOP,2013)

Test pieces was cut from the leather and placed on a flexometer . Two ends of the leather specimen was folded and gripped on each end, then subjected to 20000 flexes, after which sign of cracking or peeling was noted.The elongation caused by the specified load was calculated as shown in equation: Elongation at break = length at break - initial length

Tearing Load (INESCOP,2013)

A hole was punched on the long axis of the leather specimens. A steel rod load was passed through the hole; a force was applied until the leather torn . The tearing load was calculated shown using: Tear load(N/mm) = breaking load / mean thickness of specimen

Instant Lastometer or Grain burst(INESCOP,2013)

Circular leather pieces were cut and placed on instant lastometertester at the bottom of the circular die. The pump on the recorder was released instantly to exert pressure at the centre of the leather piece until a cracking noise on the leather was heard.

IV. RESULTS AND DISCUSSION

The physico-chemicalproperties of Neem seed oil, Neem seed oil fatliquor and Palm fatliquor oil are shown in table1:

Properties	Neem oil	Neem oil fatliquor	Palm oil fatliqour ((control)	Standard value for fatliquor(BIS,2008)
Appearance	Light yellow liquid	Brown liquid	brown liquid	Dark brown
Solubility in water at (25°C)	Insoluble	Soluble	Soluble	soluble
Odour	Strong garlic	Strong garlic	odourless	odourless
Specific Gravity at (25°C) (g/cm^3)	0.908	0.861	1.029	
pH of 10% solution	6.5	6.2	7.0	6.5 - 7.5
Fatty matter (%)	91.04	60.9	70.1	
Saponification value (mgKOH/g)	185	187	176	
IodineValue (KOH/g)	86.5	80.5	81	
AcidValue (mgKOH/g)	38 .6	30.3	27.5	
SO_3 content (%)	--	4.1	5.1	≥ 1.8
Ash content (%)	--	3.2	4.8	4 - 5

Table 1:The physico-chemical properties of neem seed oil, sulphatedneem seed oil and sulphated palm oil and standard

From the table it can be shown that the appearance of neem seed oilfatliquor and sulphated palmoil are distinct.The two fatliquor are soluble in water at 20°C giving grey . Specific gravityis higher in case of sulphatedpalm oil.The fatliquor was free from rancid or putrefactive odour of the oils. Thus the physical properties of neem seed oilfatliquor are inagreement with the standard values.

Solution	Paml oil fatliquor(control)	colour	Neem oil fatliqour	colour	standard
5% NaCl	stable	opaque	stable	translucent	Translucent/ opaque
5% Na_2SO4	stable	opaque	stable	translucent	Translucent/ opaque
5% Basic Chromium Sulphate	stable	opaque	stable	translucent	Translucent/ opaque
5% MgO	stable	opaque	stable	translucent	translucent/ opaque
5% Formic Acid	Stable	opaque	Not stable	translucent	Translucent/ opaque

Table-2: Stability in tanning solution

From Table2 , it can be shown that the prepared fatliquor emulsion is stable in salts, tanning and basificationagent as much as the control. This makes it possible for use in fatliquoring. The stability of the emulsion is due to the present of sulphate group.An emulsion may beeither transparent or opaque; this may dependent on the degree of sulphonation and neutralization. In general the opaque types of emulsion has higher degree of sulphonation.

Strength/Physical properties

The strength properties, tensile strength, tear strength were tested using an instrontensile tester while grain crack and grain brust was tested using lastometer.of two matched side leather those were fat liquoredby using sulphatedKaranja seed oil and imported sulphated castor oil have been compared which is shown inthe

Properties	Neem oil fatliquor	Palm oil fatliquor	Standard value (INESCOP,2013)
Tensile strength (N/mm^2)	14.15	19.03	≥ 15
Elongation at break (N/mm^2)	49.0	56.04	≥ 40
Tear resistance (N)	61.01	67.0	≥ 50

From the strength or physical properties analysis, it is evident that the leather fatliquored withneemseed oilfatliquor is comparable with commercial palm oil fatliquor.Some difference was observed in the physical test of leather however, results were found under thelimit of standard specification which confirmed [6], [8] the suitability of prepared fatliquor. The prepared fat-liquor possesses goodpenetration power and emulsion stability.Tensile strength is the value of load the sample cross section can bear when load by the axial load andit is related with the leather sample state of collagen fibers. Therefore, the tensile strength can characterize theflexibility of fibers beside for the strength of collagen fibers.Flexibility is one of the most important properties of leather achieved from fat-liquor. Fat-liquor reducethe friction between fiber is mainly related to the nature of sulfated oil and the quality of introduced oil. Theelongation at break can characterize the softness, flexibility [5], [6], strength and toughness of leather matrix.

Physical testing and Hand Evaluation of leather

Experimental and control crust leather were assed for fullness, softness, grain tightness (break) by handand visual examination. The leathers were rated on a scale of 0-10 points for each functional property byexperienced leather technologist, where higher points indicate leather property

Organoleptic properties

Leather made with neemseed oil and imported palm oil were analysed for their organolepltic properties which are shown in the Table.

Table-: **Organoleptic properties**

Properties	Neem oil fatliquor	Palm oil fatliquor(control)	Standard value (INESCOP,2013)
Softness	3.5	4.5	5
Fullness	4.0	4.5	5
Surface touch	4.5	4.5	5
Grain tightness	4.5	4.5	5
Odor	Odorless	Distinct	Not specified
colour	brown	brown	

From the Table it can be shown that the leather made with the neemseed oilfatliquor has thesimilar softness, fullness, touch and grain tightness, with the imported fatliquor. Leather said to be full if fiber sickening of thetanned fiber structure is well avoided.Firmness or looseness of the grain seems also be coupled with the amount of fat [12], [13] in thegrain.If the papillary layer is externally softened the grain may become loose and poor break occurs. For fullness,firmness is more critical issue; it requires proper adjustment of combination of fatliquor, dyeing andsubsequentmechanical operation.

V. CONCLUSION

The leather processed using the neem oilfatliquor and conventional imported palm oil fatliquor are comparable in physical, and organoleptic properties. It is therefore concluded that sulphatedneem oilfatliquor is suitable for fatliquoringof goat skin leather. The raw material (neem seeds) are available locally, cheap, non-toxic and nonedible. It could be a substitute for imported fatliquor.

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