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Sulphated Fatliquor Extraction from Nagkesar (Mesua Ferrea) Seed Oil for Leather Processing

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ABSTRACT
Fatliquor improves organoleptic and strength properties of leather. Fatliquor is made from non-edible *Mesua ferrea* seed oil using the sulphation process by adding sodium hydroxide to fix its pH at 5.0. FT-IR analysis confirmed that strong chemical bonding between oil & sulphonic acid group. Physico-chemical properties of sulphated fatliquor were analyzed and compared with conventional fatliquor. Sulphated *Mesua ferrea* oil fatliquor revealed to Acid value (35.43), Saponification value (255), Iodine value (76.14), Fatty matter (60.9%), soluble in water (25^0C), P^H :6.1 in 10% solution, stable against salt, tanning and basification agent, odorless and dark brown liquid. Prepared *Mesua ferrea* seed oil fatliquor is consistent in physico-chemical properties with standard value of fatliquor. As prepared eco-friendly and cost effective fatliquor may reduce dependency on edible oils for conventional fatliquor. Therefore, fatliquor made from Soxhlet-extracted *Mesua ferrea* seed oil could be used in place of traditional fatliquor in leather processing.

Keywords: Sulphated fatliquor, Soxhlet extraction, *Mesua ferrea* seed and Strength properties

1. Introduction

Raw hides/skins are transformed into valuable leather through beamhouse, tanning, post-tanning and finishing operations. Post-tanning operations e.g., neutralization, retanning, dyeing, fatliquoring use water to give leather texture and structural properties (Hansen et al. 2020). Fatliquoring, a process of coating the surface fibers or fibrils of leather with a thin layer of oil/fat (Kamely, 2021). Leather is treated with oils, fats, greases in order to improve its softness, stretchiness, elasticity and fullness by lubricating the fibers to enable them to slide over one another (Covington, 2009). As part of the emulsification process, sulphite groups are added to the structure of oils and fats to create fatliquors, which are surface active softening agents (Affiang et al. 2018). Fatliquor effects on leather's organoleptic characteristics such as softness, surface touch, odor, grain tightness, color, grain break, surface feel and on the physical properties such as tensile strength, stitch - tearing resistance, abrasive resistance of the leather.

Traditional ingredients such as fish, animals, edible vegetable oil, mineral oil etc. have been utilized to make fatliquor across a wide range of time (Covington, 1993). vegetable oils, including coconut oil, rice bran oil, olive oil are used for producing fatliquor. These oils are edible and mostly used for cooking. Most of the fish, vegetable oils are edible oil and used by the human (Quadery et al. 2015). Due to the presence of lengthy and unsaturated carbon chains, fatliquor prepared from vegetable and fish oils increases the suppleness of leather. Non-edible oil, such Nagkesar (*Mesua ferrea*) seed oil, might be a preferable alternative, nevertheless, considering the increasing price of edible oils and the necessity to create eco-friendly leather goods.

Different oils e.g, neem (Affiang et al. 2018); castor (Tawfik et al. 2017); karanja (Quadery et al. 2015) seeds oils are introduced by many researchers to produce fatliquor. All these oils are not available in our country. Bangladesh is a huge source of non-edible *Mesua ferrea* seeds which can be plant in rail and road side areas of Bangladesh. *Mesua ferrea* seeds are available in Bangladesh which are not potentially used (Sayeed et al.2004). Nagkesar is a beautiful evergreen tree and has been used in Ayurveda for centuries. For numerous Ayurvedic medicines, many parts of the plant, including the root, bark, leaves, seeds, flowers, and wood, might be employed. Nagkesar seed oil is used worldwide as medicine. It has not been used yet for making fatliquor. Though it has effective quality for producing fatliquor.

The seeds of the Nagkesar plant are processed to yield non-edible vegetable oil known as "Nagkesar oil" . *Mesua ferrea* oil contains a larger percentage of oleic acid which is appropriate for making fatliquor (Hasan, 2017). In this work, oils are extracted from *Mesua ferrea (*Nagkesar) seeds to prepare sulphated fatliquor for producing eco-friendly leather articles**.**

2. Materials and Methods

2.1 Seed collecting and processing

Mesua ferrea seeds had been collected in Rajshahi Division, Bangladesh. The seeds were taken out of the fruits, manually deshelled, then water-washed multiple times to get the sticky substances off. The seeds were then sun-dried for ten to fifteen days. The seeds were then further dried at 105°C for 12 hours to achieve a constant weight. After that, the dried seeds were crushed into a fine powder and used for extracting oil.

2.2 Extraction of oil from *Mesua ferrea* Seed

By employing the Soxhlet equipment and ethanol as solvent, oil was obtained for 24 hours. 20 gram of seed powder was taken in a small bag of thin white cloth and tied

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up with white thread. Then powdered sample was treated with 250 ml ethanol at 70-80^{0}C. After doing five cycles in Soxhlet extractor, bag was taken out and solvent-oil mixture was collected. The solvent and oil content were separated by keeping the solution overnight in separating funnel.

2.3 Physico-chemical properties of sulphated fatliquor

2.3.1 Determination of yield of oil (%)

20 g powdered seed material was treated with ethanol as solvent in Soxhlet extractor. Then oil-solvent solution was kept in a separation funnel for overnight. The percentage of oil yield was computed using this formula:

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

2.3.2 Determination of Acid value of *Mesua ferrea* oil

In a dry conical flask, 2 g of oil was added, followed by 25 ml of absolute ethanol, 2–3 drops of phenolphthalein, and heating with shaking at 60–65% for 10 minutes. After cooling, the solution was titrated against 0.1 N KOH until the pink color appeared. Finally, the formula was applied to compute the acid value:

Acid value = (Volume of KOH × N × 56)/ Weight of sample

Where, N = Normality of KOH

Free fatty acid (%FFA) = acid value × 0.503

2.3.4 Determination of Iodine Value of *Mesua ferrea* oil

In a 250 ml conical flask, 0.25 g of oil was placed along with 10 ml of chloroform and 30 ml of hanus solution and fully sealed the flask using the parafilm. After 30 minutes of stirring, 10 ml of 15% potassium iodide solution was added. 100 ml of distilled water was included as well as the iodine solution was titrated against the 0.1 N sodium thiosulfate solution till the yellow color was appeared. Then, 2-3 drops of starch solution were added, and the titration was continued until the blue color vanished. The identical process was used for blank. This formula was used to determine the iodine value:

Iodine value = {(B-S)×1.269}/weight of sample(g)

Where, B = volume of $Na_2S_2O_3$ for blank

S = volume of $Na_2S_2O_3$ for sample

2.3.4 Determination of Saponification Value of *Mesua ferrea* oil

A 250 ml conical flask containing 2 g of oil and 25 ml of an alcoholic potassium hydroxide solution was used. The flask was then heated continuously on a boiling water bath for one hour while being sometimes shaken. When the mixture was still hot, three drops of phenolphthalein were added, and the surplus potassium hydroxide was titrated with 0.5 hydrochloric acid. Same procedure was followed the like above for determination of without sample. This formula was applied to compute the saponification value:

Saponification value = {56.1×(B-S) ×N of HCl}/Gram of sample

Where, N = Normality of HCl, B = volume of HCl for blank, S = volume of HCl sample

2.4 Synthesis of *sulphated fatliquor* by Sulphation

20 g of Mesua ferrea seed oil was stirred at 18–20 °C while 4 ml of concentrated sulfuric acid and 2 ml of phosphoric acid mixture were added dropwise. The reaction was carried out slowly for 3 hours. After being agitated with 40 ml of 10% $NaCl_2$ solution, the sulphated material was placed in a funnel and left overnight. In order to prepare fatliquor, the bottom layer was removed. By adding 20 ml of a 30% sodium hydroxide solution to the fatliquor to adjust the pH 5.0, the prepared fatliquor was collected and kept at room temperature.

3. Results and Discussion

3.1 Analysis of Physical and chemical characteristics of *sulphated fatliquor*

Table1. exhibits the physical and chemical characteristics of Castor oil and *Mesua ferrea* seed oil.

Table 1. Physico-chemical attributes of *Mesua ferrea* oil and comparison with Castor oil

Properties	*Mesua ferrea* seed oil	Castor oil
Appearance	Dark reddish liquid	Pale yellow
Odor	Acrid	soft
P^H of 10% solution	6.5	6.6
Solubility in water (25^0)	insoluble	insoluble
Fatty matter (%)	93.264	73.3
Acid value	17.92	4
Saponification value	269.28	186.5
Iodine value	86.29	88

Castor oil and *Mesua ferrea* seed oil exhibit different appearances, as may be shown in table 1. These oils are insoluble in water (25^{0}C). Because of *Mesua ferrea* seed oil has a high percentage of fatty content, it can be used to make fat liquor. The amount of unsaturated fatty acids in the oil is indicated by the iodine value which is used to evaluate how unsaturated the fatty acids are. The major fatty acid in this oil, which is suited for making fat liquor, is said to be oleic acid. The greater acid value of Nagkesar seed oil makes it a desirable ingredient for fat liquor.

3.2 Analysis of Physico-chemical properties of Sulphated *Mesua ferrea* seed oil fatliquor

The physical and chemical properties of *Mesua ferrea* seed oil fatliquor and standard values of fatliquor are stated in Table 2.

Table 2 Physico-chemical properties of sulphated fatliquor and comparison with standard value

Properties	Sulphated fatliquor	Standard value (BIS 2008)
Appearance	Dark brown liquid	-
Odor	Odorless	Odorless
P^H of 10% solution	6.1	6.5-7.5
Solubility in water (25^0)	soluble	Soluble
Fatty matter (%)	60.9	-
Acid value	35.43	-
Saponification value	255	-
Iodine value	76.14	-

The iodine value of the raw *Mesua ferrea* seed oil falls following sulphation, but the acid value increases, indicating the hydrolysis of fatty molecules, according to table 2. *Mesua ferrea* seed oil was discovered to have a pH that was within the acceptable range.

3.3 Stability of Sulphated *Mesua ferrea* seed oil fatliquor

Table 3 illustrates the obtained fat liquor's consistency in various solutions.

Table 3 Stability of fatliquor in tanning solution

Solution	Sulphated fatliquor	Colour	Standard value (BIS 2008)
5% NaCl	Stable	Opaque	Translucent /Opaque
5% Na_2SO_4	Stable	Opaque	Translucent /Opaque
5% Basic Chromium Sulphate	Stable (1-2 hours)	Opaque	Translucent /Opaque
5% MgO	Stable	Opaque	Translucent /Opaque
5% Formic acid (HCOOH)	Not Stable	Opaque	Translucent /Opaque

The produced fat liquor is stable in salts, tannins, and basification agents, as demonstrated in Table 3. Because of this quality, produced fatliquor could be used in leather manufacturing. The presence of the sulphate group gives the emulsion its stability. The degree of neutralization and sulphation determines the transparency or opaqueness of an emulsion. Generally, opaque emulsion types are considered better in fatliquoring due to higher degree of sulphation.

3.4 FT-IR Analysis

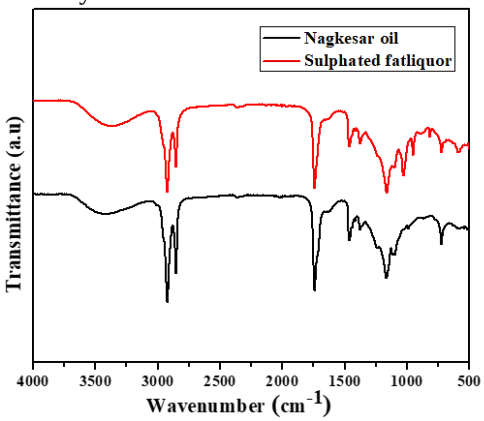

Fig.1 FTIR analysis of Nagkesar oil and sulphated fatliquor

Table 4 Analysis of FT-IR Spectra (Theivandran et al. 2015)

Nagkesar oil		Sulphated fatliquor	
Wavelength (cm^{-1})	Functional group	Wavelength (cm^{-1})	Functional group
1135	Alkyl amine	718	C-H
1737	Ketones	814	Aromatic compound
2850	Carboxylic acid	946	N-H
2926	C-H	1034	Alkyl amine
3410	N-H, C-H, O-H	1156	SO_3
		1733	Ketones
		2353	C-N
		2919	Carboxylic acid
		3376	N-H, C-H, O-H

The presence of C-H and -SO_3 group are indicated by the wide peaks at 3154 cm^{-1} and 1165 cm^{-1}, respectively. Due to the presence of N-H and Alkyl amine groups, two new peaks are detected at 930 cm^{-1} and 1027 cm^{-1}. FT-IR analysis provides confirmation on sulphation of *Mesua ferrea* oil.

4. Conclusion

Prepared *Mesua ferrea* seed oil fatliquor is consistent in physico-chemical properties with the standard value of fatliquor. FTIR analysis provides confirmation on sulphation of *Mesua ferrea* oil. The presence of higher percentage of fatty matter content confirms that *Mesua ferrea* seed oil is suitable for preparing fatliquor. Also Prepared *Mesua ferrea* oil fatliquor has stability against salt, tanning and basification agent. *Mesua ferrea* seeds are easily accessible, inexpensive, non-toxic, and non-edible. It might be an alternative for commercial fatliquor and Nagkesar seeds could be regarded as a significant source of fatliquor ingredients. It is therefore concluded that sulphated *Mesua ferrea* seed oil fatliquor is suitable for leather processing.

5. Recommendation

It is highly recommended that application of *Mesua ferrea* seed oil fatliquor on leather to check the effect and characterization of fatliquored leathers.

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7. References

[1] Hansen, É., de Aquim, P. M., Hansen, A. W., Cardoso, J. K., Ziulkoski, A. L., & Gutterres, M. (2020). Impact of post-tanning chemicals on the pollution load of tannery wastewater. *Journal of Environmental Management*, *269*, 110787.

[2] Kamely, N. (2021). “Fatliquors” for leathers: an application of microemulsion—a review. *Polymer Bulletin*, 1-26.

[3] Covington,A.D and covington (2009). Tanning chemistry-The science of leather. Cambridge, UK: Royal society of chemistry, UK, PP. 392-419.

[4] Affiang S.D., Ggamde G., Okolo V.N., Olabade V., Jekada J.Z., (2018). Synthesis of Sulphated-Fatliquor from Neem (Azadirachta Indica) Seed Oil for Leather Tannage. American Journal of Engineering Research (AJER). Volume- 7, 215-221

[5] Covington A.D., Alexander K.T.W. (1993). The preparation of soft leather part1: The mechanism of chemical softening by lubrication. Journal of the American leather Chemists association. 88(7): 24-53

[6] Quadery A.H., Uddin M.T., Azad M.A.K., Chowdhury M.J., Deb A.K., Hassan M.N. (2015). Fatliquor preparation from Karanja seed oil (Pongamia pinnata L.) and its application for leather processing. IOSR-JA. volume-8, 54-58.

[7] Tawfik H.M., Gasmelseed G.A., Mohammed F.E.F. (2017). Using Characterization and Synthesis of Fatliquor from Sudanese Castor oil. IJESRT, DOI:10.5281/zenodo.266664, ISSN:2277-9655

[8] Sayeed M.A., Ali M.A., Khan G.R.M.A.M., Yeasmin M.S., (2004). Physico-chemical characteristics of Mesua ferrea seed oil and nutritional composition of its seed and leaves. Chemical society of Ethiopia. 18, 157-166. DIO: 10.9790/5736-08115458