مجلة ساحل المعرفة للعلوم الإنسانية والتطبيقية
Sahel Almarifah Journal of Humanities and Applied Sciences
تصدر عن الاكاديمية الليبية فرع الساحل الغربي
المجلد الثاني–عدد خاص-S1
الصفحات (E-79 - E-98)

استخلاص اللانولين من صوف الأغنام الليبية، وتوصيفه الكيميائي، وتصميمه الأولي لتوسيع نطاق إنتاجه: سبيلٌ لاستغلال المنتجات الثانوية الحيوانية غير المستغلة

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الملخص

يُعَدُّ تثمين المخلفات الزراعية ومخلفات الثروة الحيوانية عنصرًا أساسيًا في التحول نحو اقتصاد دائري مستدام. ويُعد اللانولين (شمع الصوف)، وهو مزيج معقد من الإسترات، والأحماض الدهنية، والكحوليات عالية الكتلة الجزيئية، من المنتجات الثانوية القيّمة لصناعة صوف الأغنام، حيث يُستخدم على نطاق واسع في التطبيقات الصيدلانية والتجميلية والصناعية. وعلى الرغم من أهميته الاقتصادية العالمية، لا يزال الاستخلاص التجاري للللانولين في المناطق ذات الكثافة العالية من الثروة الحيوانية، مثل ليبيا، محدودًا وغير مستغل بالشكل الأمثل.

تهدف هذه الدراسة إلى استخلاص اللانولين من صوف الأغنام الليبي الخام، وتوصيفه كيميائيًا، بالإضافة إلى وضع تصور مبدئي للتدرج نحو التطبيق على النطاق الصناعي. أُجريت دراسة مقارنة على النطاق المختبري لتقييم كفاءة طريقتين للاستخلاص: الاستخلاص الساخن باستخدام الغسل المائي متبوعًا بالاستخلاص السائل–السائل باستخدام الهكسان(n-hexane) ، والاستخلاص البارد. وقد أظهرت نتائج الاستخلاص الساخن المُحسَّن أعلى مردود، حيث بلغت نسبة استرجاع اللانولين 16.69% (وزن/وزن)، وهي قيمة تقع ضمن الحدود المثلى المعتمدة صناعيًا.(5–25%)

أُجري التوصيف الكيميائي للللانولين المستخلص باستخدام تقنية كروماتوغرافيا الغاز المقترنة بكاشف التأين اللهبي(GC-FID) ، وأظهرت النتائج أن التركيب يغلب عليه مكونات إستيرية أليفاتية طويلة السلسلة، حيث سُجِّلت أعلى النسب عند الكربونات C29 (21.8%)و C30 (18.9%)و C32 (59.2%). كما أُكدت الطبيعة الأمفيفيلية (المحبة للماء والدهون) للللانولين باستخدام مطيافية الأشعة تحت الحمراء بتحويل فورييه(FTIR) ، من خلال ظهور نطاقات امتصاص مميزة لمجموعات الهيدروكسيل (3414 سم$^{-1}$)، وسلاسل الكربون الأليفاتية (2925–2855 سم$^{-1}$)، ومجموعات الكربونيل الإستيرية (1736 سم$^{-1}$).

إضافةً إلى ذلك، تم تطوير تصميم هندسي مبدئي لوحدة تجريبية بطاقة إنتاجية تبلغ 10,000 كغ/سنة، شمل حسابات موازنة الكتلة والطاقة، ومتطلبات القدرة للمحرّك الخلّاط (4.53 كيلوواط)، بالإضافة إلى تصميم المبادل الحراري. تؤكد النتائج المتحصل عليها

الجدوى التقنية لإنتاج اللانولين محليًا، كما توفر إطارًا قابلًا للتوسع وذا جدوى اقتصادية لتحويل مخلفات الثروة الحيوانية إلى منتجات حيوية عالية القيمة.

الكلمات الدالة: استخلاص اللانولين، التدرج في حجم العملية (التوسيع الصناعي)، تثمين المخلفات الزراعية، مطيافية الأشعة تحت الحمراء بتحويل فورييه(FTIR) شمع الصوف.

Extraction, Chemical Characterization, and Preliminary Scale-Up Design of Lanolin from Libyan Sheep Wool: A Pathway to Valorizing Underutilized Animal By-Product

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Abstract

The valorization of agricultural and livestock byproducts is a critical component of the transition towards a circular economy. Lanolin (wool wax), a complex mixture of esters, fatty acids, and high-molecular-weight alcohols, is a valuable byproduct of the sheep wool industry, widely utilized in pharmaceutical, cosmetic, and industrial applications. Despite its global economic importance, commercial extraction in regions with significant livestock populations, such as Libya, remains underexploited. This study investigates the extraction, chemical characterization, and preliminary industrial scale-up of lanolin from raw Libyan sheep wool. A comparative laboratory-scale study was conducted to evaluate the efficacy of hot aqueous scouring followed by liquid-liquid extraction (using n-hexane) versus cold extraction. The optimized hot extraction process yielded a lanolin recovery of 16.69% (w/w), aligning with optimal industrial benchmarks (5–25%). Chemical characterization via Gas Chromatography-Flame Ionization Detection (GC-FID) revealed a predominantly composed of long-chain aliphatic ester-related components, specifically C29 (21.8%), C30 (18.9%), and C32 (59.2%). Fourier Transform Infrared (FTIR) spectroscopy confirmed the amphiphilic structural amphiphilic nature of the extract, highlighting characteristic

absorption bands for hydroxyl groups (3414 cm^{-1}), aliphatic carbon chains (2925–2855 cm^{-1}), and ester carbonyl groups (1736 cm^{-1}). Furthermore, a preliminary engineering design for a 10,000 kg/year pilot plant was developed, encompassing mass and energy balances, agitator power requirements (4.53 kW), and heat exchanger sizing. The findings demonstrate the technical feasibility of localized lanolin production, offering a scalable, economically viable framework for converting livestock converting livestock by-productsinto high-value bio-based products.
Keywords: Agricultural waste valorization, FTIR spectroscopy, Lanolin extraction, Process scale-up, Wool wax.

Introduction

The increasing global need for natural, sustainable, and renewable sources in the pharmaceutical, cosmetic, and chemical industries has led to extensive research in the valorization of agricultural and animal wastes [20]. Among these bio-based wastes, sheep wool is found in large quantities but is often not sufficiently exploited. In the preliminary stages of treating raw wool, lanolin, a waxy yellowish substance, is secreted by the sebaceous glands of wool-producing animals in order to protect the wool and skin from environmental degradation [7]. This crude lanolin makes up 5% to 25% of the total weight of fresh shorn wool and is found to be a highly cost-effective waste material, which if untreated in textile scouring wastes, has significant adverse effects on the environment due to its high chemical oxygen demand (COD) and its inability to degrade [2, 15].

Chemically, lanolin is a complex mixture of semi-polar compounds consisting mainly of high-molecular-weight wax esters (approximately 90-95%), as well as fatty acids, sterols, diesters, and long aliphatic chain alcohols [19]. Its amphiphilic character, conferred by the presence of both polar hydroxyl (-OH) and ester (-COO-) groups attached to non-polar long hydrocarbon chains, imparts lanolin with excellent emulsifying properties, allowing it to absorb twice its weight in water. Consequently, high-purity lanolin and its derivatives are now essential for the formulation of

moisturizing cosmetics, dermatological ointments for wound healing, rust inhibitors, and industrial lubricants [6, 10].

The traditional processes used for lanolin extraction have evolved dramatically from crude water washing to highly mechanized processes. Currently, aqueous scouring at high temperatures (60-80°C) in the presence of detergents and alkalis followed by centrifugal separation is the most commonly used method for lanolin extraction. Although traditional scouring processes are effective for large-scale operations, these processes are highly polluting. Over the years, many advanced methodologies have been explored for lanolin extraction to increase yields and sustainability. For instance, studies have confirmed the efficiency of supercritical fluid extraction in producing high-purity lanolin with minimal solvent residue [5, 21]. Moreover, another study has indicated the potential for microwave-assisted extraction and green solvents for improving efficiency and reducing environmental impact [4, 13]. However, these advanced processes are extremely costly and hence beyond the reach of many developing countries and small and medium-scale enterprises.

Despite the presence of a significant livestock population with large numbers of sheep in Libya, the country is completely devoid of the process of commercial lanolin production [8]. The raw wool produced in the country as a by-product of the meat and agricultural industries is often wasted or under-processed. It is thus essential to address the gap between the current situation and the potential of the country in the production of lanolin. It is in this context that the present study is conducted with the objective of investigating the extraction and chemical characterization of lanolin from the raw wool of Libyan sheep. The objectives of the study include the following: (1) the evaluation and optimization of the extraction process of lanolin using hot aqueous scouring in comparison with cold extraction methods; (2) the elucidation of the chemical composition and functional group content of the extracted lanolin using Gas Chromatography-Flame Ionization Detection (GC-FID) and Fourier Transform

Infrared (FTIR) spectroscopy; (3) the development of a preliminary engineering design basis to address the technical viability of the process at an industrial scale of 10,000 kg per year..

1 literature review

The extraction, purification, and application of wool wax have been the focus of continuous scientific study, from traditional approaches toward modern, eco-friendly technologies. An overview of recent literature provides the epistemic basis for this study.

1.1 Conventional Extraction and Effluent Valorization

Conventional methods in the recovery of lanolin primarily focus on the extraction of lanolin from highly polluted wool scouring wastewater. Banyal and Saharan [3] described the basic chemistry of lanolin obtained from hot water and detergent pre-treatment methods, which has vast downstream application in the personal care industry. For the remediation of the environmental impact of textile industries, Madara and Namango [20] used conventional Soxhlet extraction to determine the quantity of wool grease present in the wastewater. The results obtained showed the quantity of recoverable wool grease to be as high as 9,000 kg from a single plant on an annual basis. Similarly, Ahmed et al. [1] demonstrated the potential of bioremediation in the extraction of lanolin from sheep wool. The maximum rate of extraction was observed to be as high as 27.94% using solvent-intensive Soxhlet extraction. Recently, Aissani et al. [2] were successful in using the obtained wool wax as an agricultural bio-stimulant. The results obtained showed an increase of 150% in the germination of Olea europaea seeds due to the wax’s capacity to stimulate the production of endogenous auxins.

1.2 Advanced and Green Extraction Technologies

In order to overcome the problems of increased processing times and large amounts of organic solvent used in the conventional methods, alternative extraction methods have been studied. López-Mesas et al. [13] compared automated Soxhlet extraction, Supercritical Fluid Extraction (SFE), and Microwave-Assisted Extraction (MAE), which showed that the alternative methods resulted in almost quantitative extraction with significant time- and solvent-saving effects. The alternative methods using supercritical or subcritical fluids have gained significant attention in recent years because of their "green" characteristics. Cygnarowicz-Provost et al. [4] used supercritical CO2 at 80°C and 380 bar to extract highly purified lanolin. Following the study of Cygnarowicz-Provost et al., Valverde and Recasens [21] suggested the mass transfer model of solid lanolin extraction using sub- and supercritical CO2 modified with 5% ethanol. In the quest for "greener" extraction methods, Bhavsar et al. [4] showed the advantage of using Cyclopentyl Methyl Ether (CPME), which is a "greener" solvent compared to hexane, while Goli and Solanki [9] suggested the enzymatic extraction of oil using amylase and protease obtained from Aspergillus flavus as an alternative to the conventional chemical scouring process.

1.3 Purification, Pesticides, and Clinical Safety

The purification of crude lanolin is thus a significant aspect, especially in pharmaceutical applications where pesticide traces from sheep dips and allergenic properties are issues of concern. Jones [11] has successfully purified lanolin using a two-solvent method involving hexane and N,N-dimethylformamide for the removal of 36 pesticide residues to undetectable levels. Lopez Mesas et aJone[4] have successfully used Gel Permeation Chromatography for the remediation of pesticide-lanolin mixtures. Margenat et al. [16] have successfully used supercritical CO2 for the extraction of pesticides like diazinon and ethion from lanolin at parts per billion levels. In addition, the clinical importance of lanolin has also been highlighted in

recent years by Lis [12], who discussed the paradoxical properties of lanolin in dermatology as an excellent emollient but also as a potential cause of allergic contact dermatitis, thus emphasizing the need for strict regulatory standards.

2 MATERIALS AND METHODS

2.1 Materials and Reagents

Raw sheep wool was directly obtained from a local farm in the Tajoura agricultural district in Libya. The sheep were naturally grazed and fed on dry fodder. Sheep shearing was done manually, with emphasis placed on the collection of wool fibers. The raw wool was mechanically cleaned by hand to remove all kinds of macroscopic particulate matter, such as thorns, plant residues, and soil. No commercial detergents were used in the pre-cleaning of the raw wool to prevent emulsion formation, as shown in Figure 1.

Figher 1 . Preparation of raw wool for cleaning.

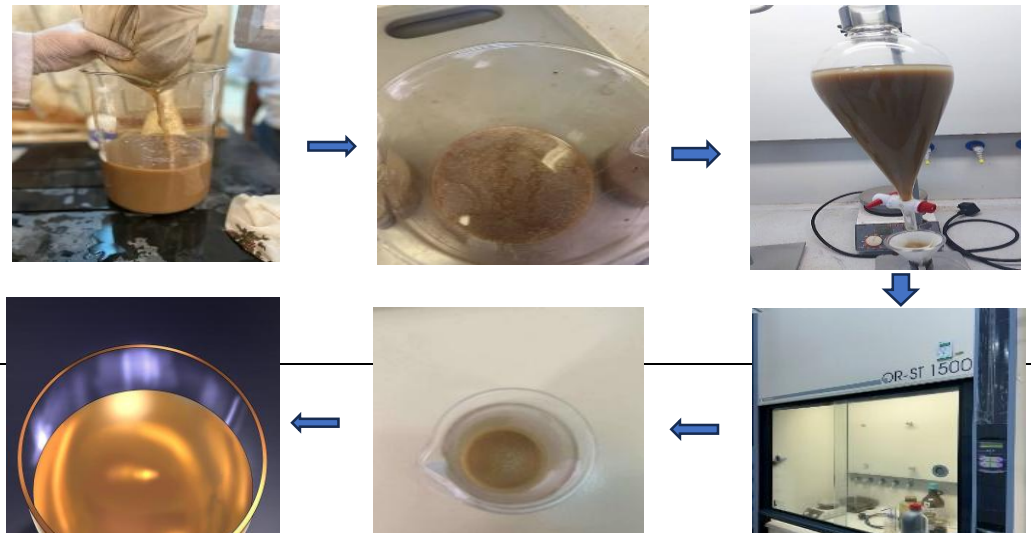
The chemicals used in the experiment were all of analytical grade. The sodium chloride used was in its raw form and was sourced locally. The primary organic solvent used in the experiment was n-hexane. This solvent had a purity of 99.0% and a molar mass of 86.18 g/mol. It was sourced from BDH Laboratory Supplies in England. Anhydrous sodium sulfate, which was used in the desiccation of the solution, was sourced from Scharlab S.L., a Spanish company. Distilled and deionized water was used in all aqueous solutions.

2.2 Lanolin Extraction Protocols

2.2.1 **HOT AQUEOUS SCOURING AND LIQUID–LIQUID EXTRACTIO:**

Initially, 250 g of pre-cleaned raw wool was precisely weighed using a precision electronic balance. The raw wool was then immersed in a 10 L vessel containing 3 L of boiling distilled water. Subsequently, 30 g of NaCl was added to the boiling solution to enhance the breakdown of cellular debris and minimize the stability of emulsion formation. The mixture was then subjected to continuous boiling and vigorous agitation for 6 hours. During the process, deionized water was added periodically to maintain a constant volume of solvent. After 6 hours of hot aqueous scouring, the hot aqueous solution was filtered using sterile medical cheesecloth to separate the exhausted raw wool. The solution was then allowed to stand for 16 hours to permit initial phase separation of CRUDE lanolin, as shown in Figure 2.

Figher 2. Preparation of wool treatment to extract lanoline.



Figher 3 . Steps of obtaining pure leonine after filtration and evaporation of hexane.

2.2.2 COLD EXTRACTION (CONTROL STUDY):

In order to evaluate the effect of thermodynamic conditions on extraction yields, a control experiment was conducted. 250 g of raw wool was immersed in 3 L of tap water and subjected to vigorous agitation. After formation of a superficial emulsion layer, 250 mL of the emulsion solution was collected and subjected to a similar liquid-liquid extraction process using 25 mL of n-hexane, as shown in Figure 4.

Figher 4. The collected sample of the cold-water experiment.

2.3 Chemical Characterization

2.3.1 GAS CHROMATOGRAPHY–FLAME IONIZATION DETECTION (GC–FID):

The carbon number distribution was determined by a VARIAN CP-3800 Gas Chromatograph, operated under a standardized protocol for ASTM D2887. A capillary column, CP-Sil 5 CB (60 m x 0.25 mm ID, 0.25 micrometer film thickness), was used. The injector was operated at 300 °C, with a split ratio of 1:200. The initial

oven temperature was set at 40 °C for 1 minute, then increased at a rate of 5 °C/min to 300 °C, and finally held at 300 °C for 60 minutes. The FID detector was operated at 300 °C. The sample solutions were made in fresh n-hexane.

2.3.2 **FOURIER TRANSFORM INFRARED (FTIR) SPECTROSCOPY: FTIR** spectra were recorded on an Agilent Technologies Cary 630 FTIR Spectrometer, equipped with an Attenuated Total Reflectance (ATR) accessory. Spectra were collected over a range of 4000-650 cm^{-1}, with a resolution of 8 cm^{-1}, accumulating 8 scans for the sample, against 8 background spectra.

3 RESULTS AND DISCUSSION

3.1 Extraction Yield and Process Efficiency

The efficiency of the lanolin recovery process was measured gravimetrically. The hot aqueous scouring procedure followed by the liquid-liquid extraction using n-hexane yielded 41.72 g of lanolin from 250 g of raw dry wool, which equaled a 16.69% (w/w) yield.

The measured yield is within the higher end of the reported range in the global literature on the subject. The reported range is between 5% and 25% depending on the sheep breed used for wool production. The reported yield is a consequence of the high temperature used in the aqueous wool scouring procedure. The high temperature significantly decreases the dynamic viscosity of the high molecular weight wool waxes.

This increases the solubility of the wool wax and the mass transfer from the wool fiber matrix to the aqueous phase. On the other hand, the cold wool extraction procedure using ambient temperature yielded a very low amount of product with significant levels of impurities. The nature of the product is indicative of the thermodynamic conditions necessary for the dissolution of the wool wax from the keratinous wool qid-xthermodynamically favorable for the dissolution of the wool

wax from the keratinous wool matrix because of the hydrophobic nature of the wool wax. Therefore, the hot aqueous procedure is a critical step in the lanolin recovery process..

3.2 Chemical Characterization

3.2.1 **GC-FID ANALYSIS FOR THE DISTRIBUTION OF** Hydrocarbons: GC-FID provides a good quantitative picture for the distribution of carbon chains that determine the physical characteristics of the lanolin. Quantitative results (Table I) indicate that the extract is dominated by saturated hydrocarbons with a significant preference for carbon chains between C29 to C32.

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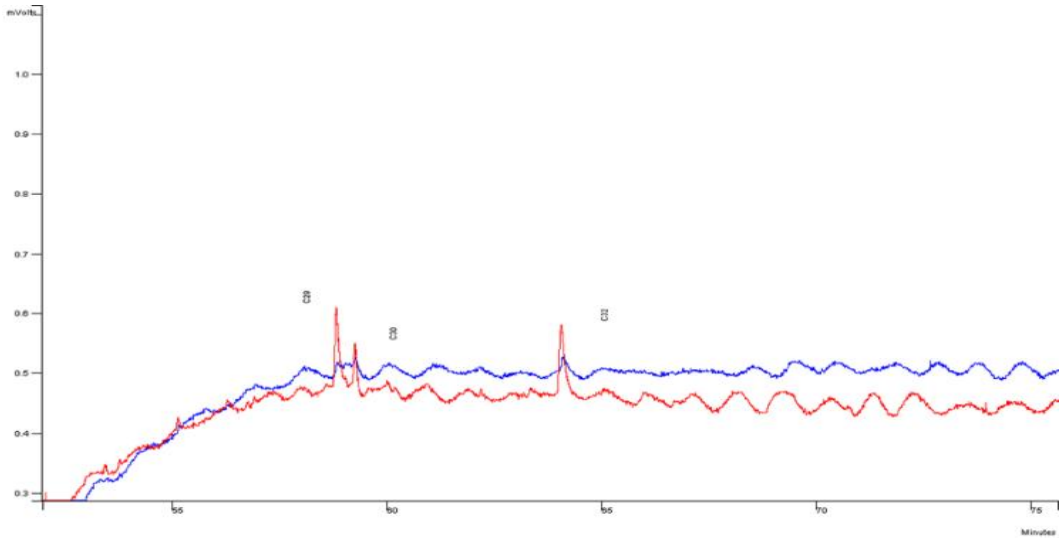
TABLE I .HYDROCARBON COMPOSITION OF EXTRACTED LANOLIN VIA GC-FID.

Composition	**Wt%**
C_{29}	21.878
C_{30}	18.933
C_{32}	59.189
Total	100.000

The dominance of C32 (59.18%) is a clear indication of the extraction of the heaviest and most stable wax esters, which are characteristic of the best quality lanolin. The presence of these heavy aliphatic chains is a direct contributor to the substance's semi-solid state at room temperatures, oxidative stability, as well as its barrier effects when used in dermatological products [5]. Of critical importance is the fact that the sample extracted using cold extraction does not have any observable peaks for the presence of long-chain hydrocarbons under the same GC-FID operating conditions, as shown in Figure 5.



Figher5.GC-FID chromatogram of hot-extracted lanolin.

4.2.2 FTIR SPECTROSCOPY ANALYSIS: TO VERIFY THE MOLECULAR structure and functional group identity of the extracted lanolin, FTIR-ATR spectroscopy was utilized. The spectrum obtained contained a variety of intense bands characteristic of a specific type of compound, as shown in Table II

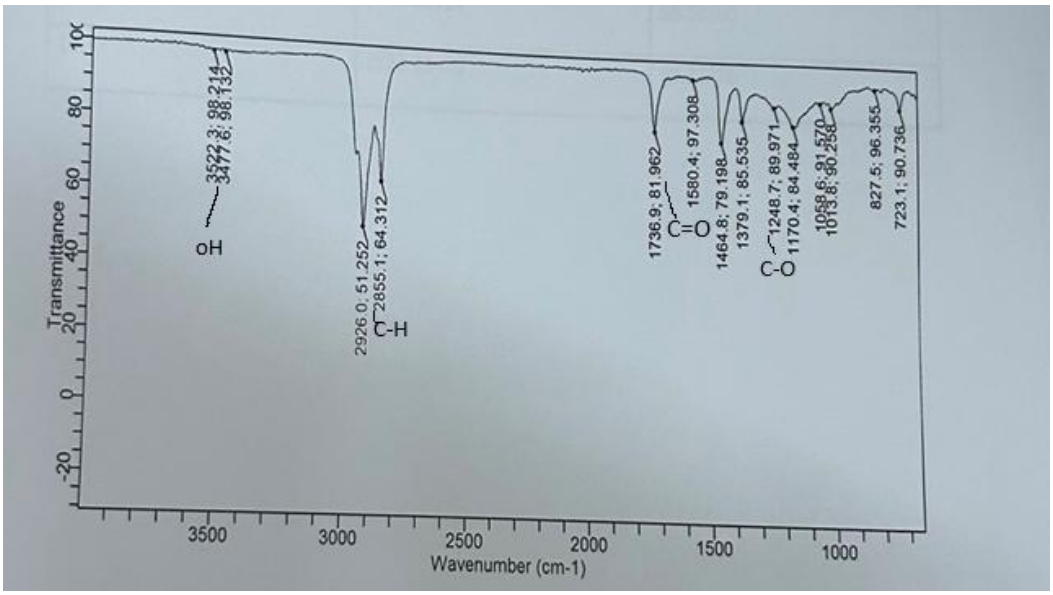
TABLE II. HYDROCARBON COMPOSITION OF EXTRACTED LANOLIN VIA GC-FID

Peak Number	**Wavenumber (cm^{-1})**	**Intensity**
1	723.10354	86.88404
2	801.37764	92.52266
3	1114.47401	84.71953
4	1174.11142	77.64703
5	1237.47616	81.94503
6	1379.115	81.46566
7	1461.11643	76.3661
8	1707.12073	79.9464
9	1736.93943	74.90644
10	2855.14078	60.45002
11	2925.9602	49.15841
12	3414.24146	97.03411

The broad, distinct absorption band at 3414 cm^{-1} is assigned to the fundamental O-H stretching vibration, which implies the presence of free high-molecular-weight lanolin alcohols and sterols that are necessary for the substance's emulsification properties [6]. The intense doublet at 2925 cm^{-1} and 2855 cm^{-1} is assigned to the asymmetric and symmetric stretching vibrations of the C-H bond in the aliphatic methyl and methylene groups, which is consistent with the GC-FID data showing extensive hydrocarbon chain presence.

Notably, the strong absorption peak at 1736 cm^{-1} is assigned to the carbonyl (C=O) stretching vibration of ester functional groups. Since lanolin contains up to 97% high

molecular weight esters of long-chain wax [23], the presence of this peak is a major chemical signature for the substance. The presence of strong hydrophobic C-H stretches with distinct O-H and C=O stretches is consistent with the substance's amphiphilic nature, as shown in Figure 6.



Figher 6.FTIR-ATR spectrum of the extracted lanolin.

5 PRELIMINARY SCALE-UP AND ENGINEERING DESIGN FRAMEWORK.

A rigorous evaluation of process scalability is necessary to successfully transfer laboratory success to industrial-scale operation. A preliminary design for the process was developed to treat a quantity of raw wool to produce 10,000 kg of purified lanolin annually.

5.1 **Mass and Energy Balances**.

With a conservative batch processing time of 16 hours for a 300-day operating year, or a total of 450 batches annually, the required lanolin production rate is 23 kg/batch. Using the yield obtained from the experiment at 16.69%, the amount of raw wool required for each batch is 137.8 kg, along with the corresponding amount of

water required for the process, which is 275.6 kg, maintaining the solid-to-liquid ratio at 1:2.

The amount of energy required to heat the aqueous solution from the ambient temperature of 25°C to the required operating temperature at 80°C was computed. Using the value for the specific heat capacity of the solution, which is approximately 4.18 kJ kg^{-1} K^{-1}, the total sensible heat required for the solution is approximately 95,042 kJ. Divided by the total batch heating period, the required net heating power is approximately 1.65 kW, which is readily achievable using saturated steam generated from an auxiliary boiler and supplied to a shell-and-tube heat exchanger.

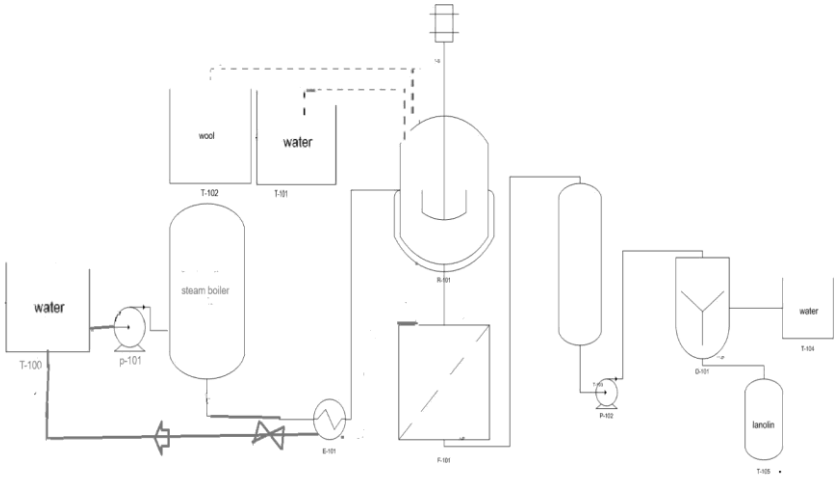
5.2 Reactor Sizing and Agitator Dynamics

The batch-wise mass input volume is the key factor for the decision-making process for a continuous stirred tank reactor or a mixing tank with a minimum operating volume of 577.85 L. Due to the high viscosity of the wool water lanolin mixture, it is important that the agitation equipment be selected with care to prevent temperature gradient buildup. A six-bladed flat turbine was selected for its performance capabilities in generating radial flow in heavy suspensions. As the tank diameter is calculated to be 1.22 m with an optimum impeller diameter of 0.77 m, fluid dynamic modeling was used. To achieve a speed of 3.23 m/s, which falls within the recommended range for medium to severe slurry mixing conditions (2.5 to 4.1 m/s), an impeller speed of approximately 80 RPM is required. Using the dimensionless power curve (Po $\approx$ 6.0 for the calculated Reynolds number regime of $7.59 * 10^4$), the motor power required for the agitator was calculated to be 4.53 kW.

5.3 Heat Transfer Sizing

The heat exchanger of the shell and tube type with saturated steam was chosen for the purpose of accommodating the heat load. A conservative value of 787.24 W/m^2 °C for the overall heat transfer coefficient (U) for the aqueous slurry was assigned.

The log mean temperature difference (ΔTlm) was also calculated and found to be 47 °C. The heat transfer surface area (A) was then determined. The calculated value is quite compact at 2.57 m², well below the standard value of 51 m². Furthermore, the calculated value for the pressure drop (ΔP) across the heat exchanger is 0.25 bar, which is well within the limits of the safety and efficiency criterion of 0.3 bar or less, as shown in Figure 7.



Figher7. Proposed Process Flow Diagram (PFD) for the industrial-scale extraction of lanolin from raw sheep wool.

6 CONCLUSION

The present study thus successfully demonstrates the chemical viability and potential for scaling up the extraction of valuable lanolin from underutilized Libyan sheep wool. The hot aqueous scouring procedure coupled with the efficiency of the liquid-liquid extraction using n-hexane was found to be highly efficient, producing a yield of 16.69% (w/w) comparable to existing international commercial standards. The critical importance of the application of elevated thermal dynamics in the extraction of the heavy wax matrix was conclusively established, as control experiments carried out at room temperatures failed to produce any measurable amounts of the product.

The chemical composition of the extracted lanolin was fully characterized, confirming the purity and structural integrity. The GC-FID results showed a distribution pattern dominated by C29, C30, and C32 carbon chains, confirming the presence of highly stable ester chains. The presence of the key ester groups was further confirmed using FTIR, which showed the presence of the ester carbonyl groups at 1736 cm^{-1} and hydroxyl groups at 3414 cm^{-1}. Furthermore, the addition of a techno-engineering design phase demonstrates the potential for scaling up the process for a 10,000 kg/year plant. The energy requirements for the process are relatively low at 1.65 kW thermal power and 4.53 kW mechanical power. The simplicity of the unit operations required for the hot aqueous scouring procedure makes the process attractive for implementation in the region. In future studies, the optimization of the pilot-scale procedure could involve the use of a green solvent system.

6.1 Acknowledgment

The authors would like to extend their sincere gratitude to the Department of Chemical Engineering, University of Tripoli, for providing the essential laboratory facilities needed to carry out this research. In addition, thanks are given to the Petroleum Research Center (PRC) and the Libyan Advanced Center for Chemical Analysis for their invaluable assistance in carrying out this work, especially in terms of executing GC-FID and FTIR spectroscopy analyses.

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