

Comprehensive view on chemistry, manufacturing & applications of lanolin extracted from wool pretreatment

Amit Sengupta* and Jagadananda Behera

Scientist at COE in Sportech Lab at Wool Research Association, Thane, Maharashtra, India

(Attached to Ministry of Textiles, Govt. of India)

Abstract: - Lanolin also called wool wax or wool grease is a yellow waxy substance secreted by the sebaceous glands of wool-bearing animals. Most lanolin used by humans comes from domestic sheep breeds that are raised specifically for their wool. Crude lanolin constitutes about 5–25% of the weight of freshly shorn wool. The wool from one Merino sheep will produce about 250–300 ml of recoverable wool grease. Lanolin is extracted by scouring the wool in hot water with a detergent to remove dirt, wool grease (crude lanolin), suint (sweat salts), and anything else stuck to the wool. The wool grease is continuously removed during this washing process by centrifugal separators, which concentrate it into a wax-like substance melting at approximately 38°C. Lanolin and its many derivatives are extracted from wool scouring liquor and converted to a value added products that are generally used extensively in both the personal care (e.g. in high value cosmetics, facial cosmetics, lip products, etc.) and health care sectors. It is frequently used in protective baby skin treatment and as a treatment for sore nipples in breastfeeding mothers. Lanolin is used commercially in many industrial products ranging from rust-proof coatings to lubricants.

Keywords: - Wool Grease, Grease Recovery, Lanolin Refinement, Applications

I. INTRODUCTION

Wool is a natural, protein, multi-cellular, staple fibre from the fleece of domesticated sheep. Wool fibre contains different contaminants such as Natural, acquired and applied. Natural impurities in the fleece consist primarily of wool grease and suint that are produced by the sheep. Wool grease is secreted from the sheep's skin and accumulates in the wool fibre as it grows. Wool grease is a mixture of fats and oils with a combined melting point of approximately 40°C. After separation during scouring, wool wax is contaminated with detergent and suint and is, therefore called wool grease, as distinct from wool wax [1]. Wool grease can be emulsified in water by the addition of a detergent. Suint is made up from a mixture of primarily potassium-based salts of fatty acids, such as oleic and stearic acids. It is excreted from the sweat glands of the sheep and dries onto the skin and wool fibres. Suint is highly soluble in water and can be isolated from the raw wool by aqueous extraction. Acquired impurities are picked up from the animal's natural environment and can be categorized as mineral or vegetable contaminants. Mineral contaminants include dirt, dust, sand and stones. These are usually quite straightforward to remove by washing. Vegetable matter (VM) caught in the animal's fleece commonly includes straw, grass, seedpods, pieces of plant tissue and twigs. Parasites such as ticks and blowfly maggots as well as faeces attached to the wool are generally defined as VM along with the true vegetable contaminants. Applied impurities cover the range of farm chemicals such as sheep dip and fertilizers applied directly to the sheep or to the pasture upon which they are grazing. These generally only occur in trace quantities and can be very difficult to separate from the other contaminants.

The use of crude wool grease washed from the fleece of sheep has been known for centuries. The ancient Greeks (about 700 B.C.) are known to have extracted it by boiling wool and skimming "fat" from the surface. Liebrich and Braun of Germany are credited with developing the first refined neutral wool grease and coining the name "lanolin" in 1882.

Table 1 Ingredients of Merino and crossbred wool [1]

Ingredients	Merino	Crossbred
Fibre	49	61
Dirt	19(6.3-43.8)	8 (7.9)
Suint	6 (2-12)	8 (2.2-12.1)
Grease	16 (10-25.4)	11 (1.6-8.5)
Water	10	12

Table 2 Typical effluent characteristics (after wool grease recovery) [2-10]

Parameters	Contaminant Heavy Scour Effluent	Rinse Water
Biological Oxygen Demand (BOD) [mg/L]	9,800 – 50,000	200 – 1,000
Chemical Oxygen Demand (COD) [mg/L]	30,000 – 100,000	500 – 2,000
Total Suspended Solids (TSS) [mg/L]	20,000 – 60,000	100 – 700
Solvent Extractable (SE) [mg/L]	1,000 – 2,000	50 – 1,500

II. CHEMICAL COMPOSITION OF WOOL GREASE

Wool Grease is a natural substance excreted from the sebaceous gland attached to the root of each wool fibre in the basal layer of the sheep’s skin [2].

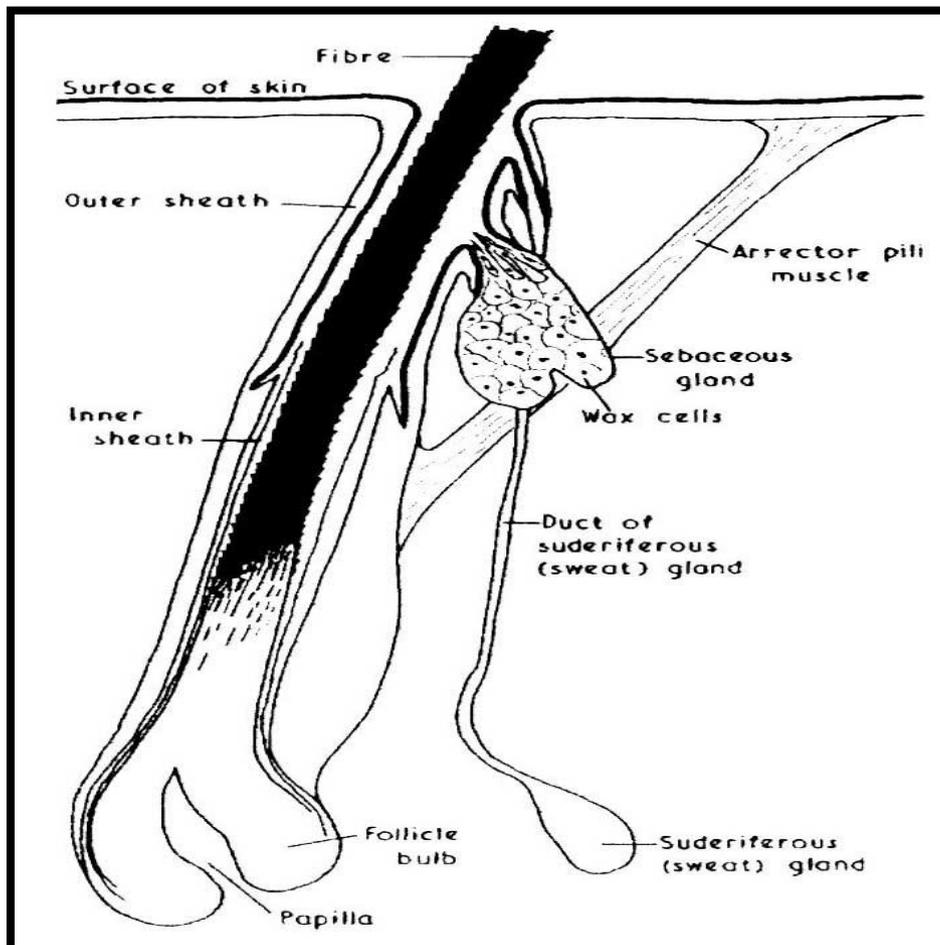


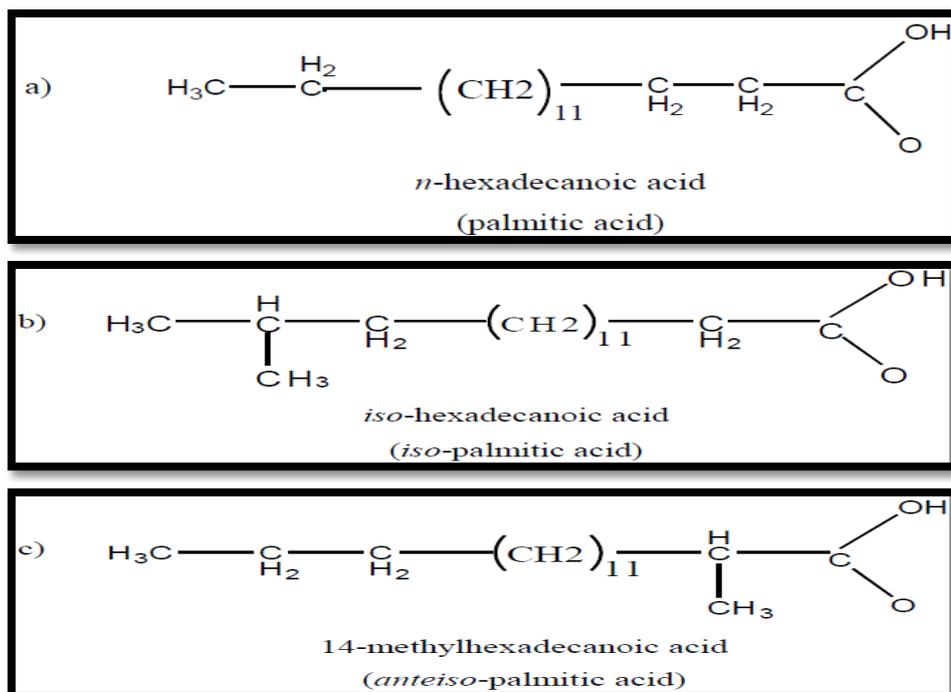
Fig 1 Dermal detail of a wool follicle

The wax excretion forms a hydrophobic coating on the fibre, protecting it from the elements. By chemical definition, the absence of glycerol in this oily excretion makes it a wax. Wool wax itself is a complex mixture of naturally occurring esters of water, insoluble alcohols and higher fatty acids.

Table 3 Physical properties of wool wax [3-10]

Physical Property		Approximate content value
Colour		Yellow to pale brown
Specific Gravity (15°C)		0.94-0.97
Refractive Index (40°C)		1.48
Melting point		35-40°C
Free acid content		4-10%
Free alcohol content		1-3%
Iodine value (Wijs)		15-30
Saponification value		95-120
Molecular weight (Rast; in Salol)		790-880
Proportion of fatty acid		50-55%
Acids:	Melting Point	40-45°C
	Iodine value (wijs)	10-20
	Mean Molecular weight	330g/mol
Alcohol:	Melting Point	55-65°C
	Iodine value (wijs)	40-50
	(Dam)	70-80
	Mean Molecular weight	370 g/mol

Isolation of specific esters is extremely difficult and seldom attempted [3-10] but detailed analysis has been carried out on the free acids and alcohols from which the esters are formed. In 1954 a large range of wool wax acids were identified and found to fall into 4 distinct series: normal (Fig a), *iso*- (Fig b) *anteiso*- (Fig c), and *hydroxy*-acids (Fig d). Examples of each series are given below:



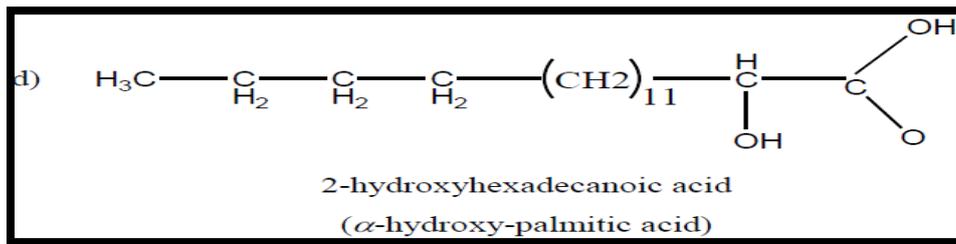


Fig 2 (a, b & c) Structure of wool wax acids

Table 4 Acidic fraction of wool wax

Acidic Fraction	Approximate content (% of total Acids)
n-acid	7
Iso-acid	22
Anteis-acid	29
α -Hydroxy-n-acids	25
α -Hydroxy-iso-acids	3
Total	86
Residual (Speculated to be mostly saturated acids)	14

The free alcohol component is primarily made up of three groups: aliphatic alcohols, sterols, and isocholesterol. A summary of these groups and their constituents is given in Table 5.

Table 5 Alcoholic fraction of wool wax

Alcoholic Fraction	Approximate content (% of total Acids)	
Aliphatic Alcohol	n-alcohol	4
	Iso- alcohol	6
	Anteis- alcohol	7
	n-Alkan-1,2-diols	0.5
	Iso-Alkan-1,2-diols	25
	Iso-Alkan-1,2-diols	3
	Sterols	28
	Isocholesterol	27
	Hydrocarbons	1
Total	78	
Unidentified Residue	22	

From a practical perspective, the wool grease dispersed in scour effluent consists of two main fractions, typically referred to as ‘oxidised’ and ‘unoxidised’ wool grease. The oxidised wool grease has been shown to be associated with the tip of the wool staple where the grease is exposed to the air and the external environment while the unoxidised fraction has been shown to be more predominant at the base and centre of the wool staple [3-10]. It is generally agreed that the oxidised fraction has a higher density than the unoxidised, but the actual values reported in various works vary widely.

III. WOOL GREASE RECOVERY

Due to its low specific gravity, unoxidised wool wax is most commonly recovered by passing the scour effluent through a centrifuge. At present, stacked disc centrifuges similar to those employed in the dairy industry are used in either a 2 or 3 stage separation and purification process. The main processes used throughout the world for wool grease recovery involve some combination of thermal cracking and centrifuging. Solvent extraction methods have been extensively developed and typically give a very high quality product [3-10], but these technologies have never gained widespread use.

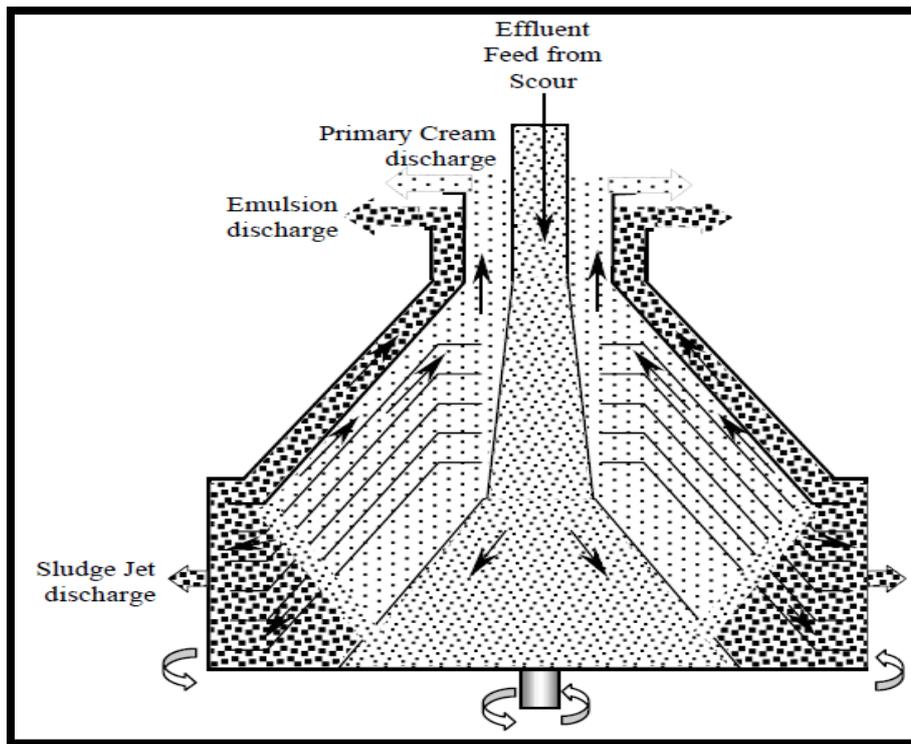


Fig 3 Primary centrifuge

3.1 SINGLE-STAGE WOOL GREASE RECOVERY

Scour effluent heated to at least 64°C is fed from above into the centre section of the primary centrifuge as shown in Figure 3. Inside the centrifuge the effluent flows into the stack of cone shaped discs, which rotate at approximately 6000rpm [1-10]. In the disc stack the lighter wool grease flows upwards between the discs towards the centre of the bowl where it is discharged as primary cream. A second emulsion phase flows downwards between the discs due to its higher density, from where it flows up the walls of the outer bowl and is also discharged at the top of the centrifuge. Any dirt and heavy solids in the feed are also carried downwards between the discs but, due to having higher density than the aqueous emulsion phase, do not flow up the walls with the emulsion phase and are continuously discharged through jets in the bottom of the bowl wall. The primary cream is then fed into a thermal-cracking tank where it is held for in excess of five hours at approximately 90°C. Over this period the cream splits into three distinct phases.

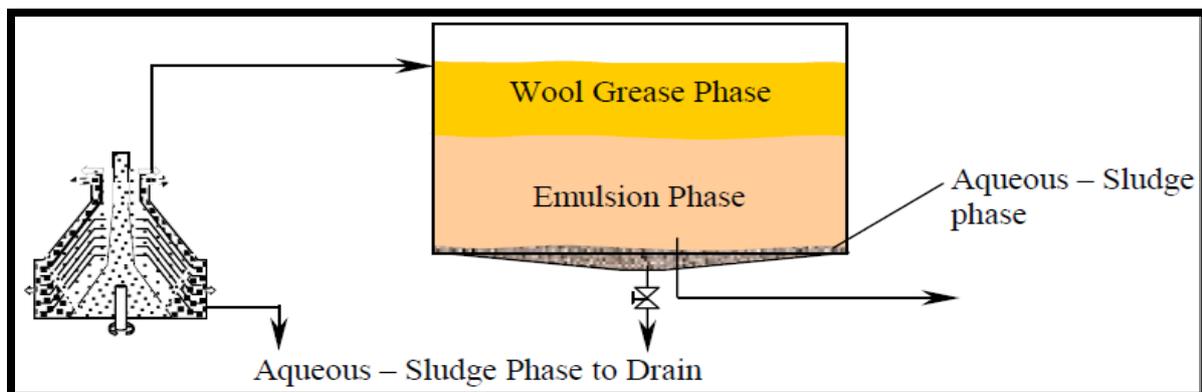


Fig 4 Single-stage wool wax recovery

After the solution has been given time to separate, the heavy aqueous phase is drawn off the bottom of the tank to drain, the emulsion phase is recycled to the centrifuge and then the wool grease floating in the top of the tank is drawn off into drums. Due to the rising value of recovered wool grease and mounting restrictions on effluent discharges, single stage wool grease recovery is no longer used in the main stream scouring industry.

3.2 TWO-STAGE WOOL GREASE RECOVERY

In a two-stage system, primary centrifuges and thermal cracking tanks are again used, but this time the cream and emulsion from the cracking tank is fed to a bank of secondary centrifuges for further purification. In a two-stage system the cream from the primary centrifuges is around 60 – 80% wool wax. The product from the secondary centrifuges is typically in excess of 99% pure. [10]

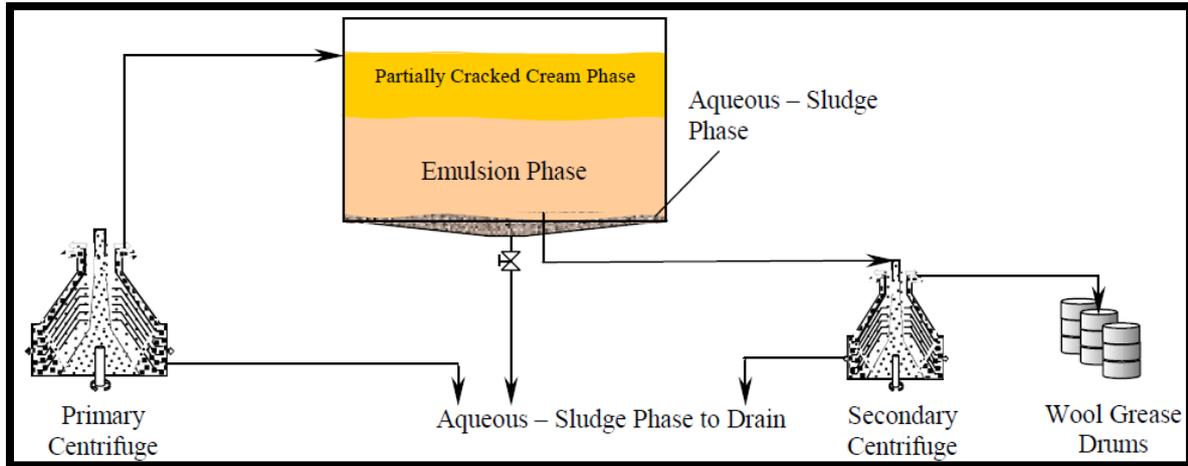


Fig 5 Two-Stage Wool Grease Recovery

3.3 THREE-STAGE WOOL GREASE RECOVERY

In a three-stage system a greater overall proportion of the wool grease is recovered from the effluent by the primary centrifuges. The resultant primary cream however is typically only 10 – 20% grease. This high volume low strength primary cream is then thermally cracked and passed through the secondary centrifuge at approximately 90°C resulting in a secondary cream containing 70– 80% wool grease. The cream from the secondary centrifuge, still at 90°C, is then passed to a purifying centrifuge where it is mixed with clean hot water (which aids in washing impurities from the grease) and is then separated to produce wool grease with purity in excess of 99%.

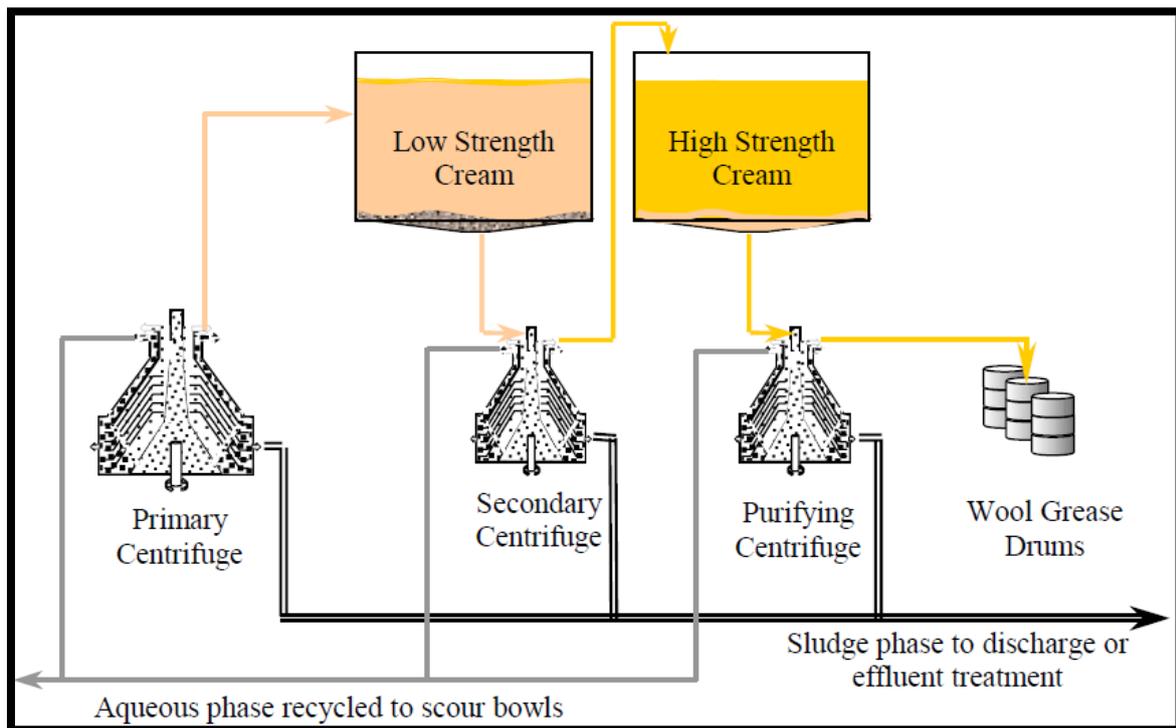


Fig 6 Three-Stage Wool Grease Recovery

IV. LANOLIN REFINEMENT

The harvested wool wax is a dark, highly viscous and greasy paste. In this crude form, it can be used in several technical applications. However, due to the colour, peculiar odour and sizeable amount of impurities it retains at this stage, the wool wax must be refined further before it can be used in cosmetic products and by the pharmaceutical industry [11, 12].

Table 6 Constituents of Lanolin Alcohols and Lanolin Fatty Acids [13]

Lanolin Alcohols	Number Identified	Contents (wt%)	Carbon Length	Carbon Length of Major Fractions
Aliphatic Mono-Alcohols				
Normal	16	1.6	C14-C34	C24,C26
Iso	11	6.5	C14-C36	C20,C22,C26
Anteiso	11	9.0	C17-C35	C21,C25,C27
Aliphatic 1,2 diols	14	0.4	C12-C2	C16,C18,C20
Normal				
Iso	9	5.9	C14-C3	C18,C20,C22,C24
Anteiso	8	2.4	C15-C29	C21,C23
Total of aliphatic alcohols	69	25.8		
C27 Sterols				
Cholesterol		38.0		
Dihydrocholesterol		trace		
Cholesta-3,5-dien-7-one		3.6		C27
C30 Trimethyl Sterols				
Lanosterol		14.8		
Dihydrolanosterol		10.3		
3-Hydroxylanosta-8-en-7-one		1.6		C30
Hydrocarbons & undetermined		5.9		
Total unsaponified matters		100.0		
Non-Hydroxy Fatty Acids				
Normal	27	12.1	C8-C38	C14,C16, C24,C26
Iso	17	22.1	C8-C40	C14,C16,C18,C20,C26
Anteiso	18	26.3	C7 -C41	C15,C19,C21,C25,C27
Hydroxy Fatty Acids				
Normal	23	21.8	C10-C32	C16
Iso	12	4.5	C12-C34	C18,C24
Anteiso	12	0.8	C11-C33	C23,C25
Hydroxy Fatty Acids				
Normal	14	3.0	C22-C36	C30,C32
Iso	8	0.8	C22-C36	C30,C32
Anteiso	7	1.3	C23-C35	C25,C31
Total	138			
Unsaturated fatty acids	42	2.1		C16,C18
Polyhydroxy fatty acids	—	4.7		—
Total fatty acids confirmed	180	99.5		

4.1 FIRST STAGES OF REFINEMENT

The techniques and materials used for refining lanolin vary from processor to processor. Generally, the first step involves the break-up of soaps and removal of acid-soluble impurities by refluxing the crude wool wax in an aqueous acid solution. After separation and disposal of the aqueous phase, the free fatty acids in the wax are neutralized via an alkaline treatment, preferably using a sodium hydroxide or carbonate solution. The resulting soaps, which are formed by the reaction of fatty acids with alkali metal ions such as sodium, can be extracted using mixtures of ethanol or isopropanol with water. The alcohols function both as a co-solvent and an agent to reduce the emulsifying properties of surfactant molecules. This makes sure that undesired materials are washed out of the wax mixture effectively.

4.2 IMPROVING THE PURITY

Depending on the desired purity grade, this extraction procedure can be repeated with more highly concentrated alcohol mixtures. A further reduction of contaminants can be achieved through treatment with adsorbents, such as activated carbon, followed by filtration. Residual pesticides and odours can be removed by exposure to high vacuum and simultaneous heating of the wool wax. This processing step also reduces the amount of free lanolin alcohols. By slightly varying vacuum and temperature conditions, their content can be reduced to less than 3% by weight, which is crucial if the desired result is product hypoallergenicity.

4.3 COLOUR CHANGE

At this stage in the refinement process, most of the impurities - free fatty acids, detergents and pesticides will be sufficiently reduced and the product will be pleasantly deodorised. The dark colour will have faded into a pale yellow tone. While the colour of the refined lanolin has nothing to do with its quality, some cosmetic applications require bright lanolin. This can be achieved by bleaching the wax with common oxidative reagent such as: hydrogen peroxide. If the lanolin is not bleached, then small concentrations of another approved antioxidant, such as butylated hydroxytoluene (BHT), are usually added at the end of the refinement process. This stops the air getting to the lanolin, preventing superficial degradation by autoxidation.

4.4 FINAL STEPS

Finally, residual water is removed through a vacuum-drying process to give refined lanolin anhydrous, a pale, ointment-like substance with a scarcely recognisable odour.

Table 7 Components of Crude centrifugal wool wax and refined pharmaceutical lanolin [13]

Component	Crude centrifugal wool wax	Refined pharmaceutical lanolin
Water	1-5% w/w	0.05% w/w
Free fatty acids	1-8% w/w	0.30% w/w
Free fatty alcohols	6-12% w/w	2.50% w/w
Wax esters	75-90% w/w	97.0% w/w
Water soluble salts of potassium and sodium	0.1-0.2 w/w	Not Quantified
Lipid soluble salts of calcium, magnesium, iron, aluminium	0.2-2.0 w/w	Not Quantified
Detergent residues	0.1-1.0 w/w	0.02% w/w
Pesticide residues	20-150 mg kg ⁻¹	0.8 mg kg ⁻¹
Polyaromatic hydrocarbons	0.1-0.5 mg kg ⁻¹	0.08 µg kg ⁻¹
Particulate insoluble matters	0.1-2.0	not quantified
Peroxide value	Not quantified	12.0 meq. kg ⁻¹
Colour	Dark brown	Pale to yellow
Odour	Peculiar smell	Almost odorless

It is soluble in organic solvents like diethyl ether, chloroform and chloroform/methanol mixtures, but poorly soluble in ethanol. It does not dissolve in (but is mixable with) water, forming stable water-in-oil (w/o) emulsions.

V. APPLICATION OF LANOLIN AND ITS DERIVATIVES

Uses of Lanolin and its derivatives are as follows [14-25]:

5.1 LANOLIN AS A PERSONAL CARE

Wool wax is a natural substance, designed by nature to soften both skin and wool fibres, and to protect them against adverse weather conditions. The best known uses of refined wool wax products (lanolin and lanolin derivatives) are in medicine, cosmetics and toiletries, which take advantage of these natural protective qualities. Lanolin is a key ingredient in some of the world's most popular cosmetics and pharmaceuticals. Without it, they would not have the emollient qualities that protect and care for our skin and hair. The composition of lanolin resembles the intercellular lipids of the stratum corneum. This is the outermost layer of the skin, which consists of cholesterol, cholesterol derivatives and free fatty acids. These lipids play a crucial role in the skin's moisture control. Under normal conditions, water continuously evaporates from the skin's surface. Insufficient rehydration from lower epidermal layers leads to a dry, inflexible and brittle stratum corneum. Anhydrous lanolin can absorb more than 200% of its weight in water (WW) to form stable water-in-oil (w/o) emulsions. It's also capable of redistributing this moisture to environments of low relative humidity [11].



Fig 7 Lanolin is also used in lipsticks and eye make-up

Lanolin is also widely used in:

- foundation creams and other skin-cream products as an emulsifier, stabiliser, emollient and skin moisturiser;
- oil-based skin lotions and cleansing oils as a skin moisturiser and to control viscosity;
- toilet soaps as a superfatting agent, minimising the dehydrating effect of detergents, and to retain perfume;
- aftershaves as a skin moisturiser and to control viscosity;
- nail polish removers to prevent the defatting of the surrounding skin;
- lipsticks and eye make-up as a film modifier and crystal inhibitor, for more uniformly dispersed pigment;
- hair dressings and shampoos, as a conditioner against drying, scaling and brittleness of the hair shaft;
- hair sprays, as a plasticiser;
- hair bleaching agents, as a pH-stable emulsifier.

5.2 MEDICAL APPLICATIONS: Lanolin is widely used in:

- ointment bases, burns dressings and wound sprays
 - as an emulsifier, stabiliser and emollient
 - to support the wound healing process
 - to deliver active ingredients through the skin (trans-dermal);
- pigmented medications (e.g. zinc oxide), as a dispersing agent;
- topical products for cutaneous infections (e.g. acne) and in deodorising toiletries, as an anti-microbial and disinfectant.
- ophthalmic ointments, as an emollient with high physiological compatibility and low irritation potential;
- suppositories substantial base, as a carrier for active ingredients;
- surgical adhesive tapes, as an impregnating agent, plasticiser and skin-suited stack enhancer;
- chewing gum bases as a food additive (physiologically compatible emollient);
- pre-blended combinations for specific purposes, such as absorption bases.

5.3 INDUSTRIAL APPLICATION

Lanolin is also used in various industrial applications such as:

▪ Anti-corrosive effect on ferrous metals

It is biodegradable and non-toxic, making it an ecologically friendly substance. Lanolin is also compatible with numerous additives that modify the consistency and characteristics of the resulting protective films (e.g. hard, soft, water soluble or insoluble). Strongly corroded seawater tanks and ships can be rust-protected with low viscous lanolin products. In the floor method, the liquids are dispersed on the weathered underground. The oil then creeps into the cavities of the rust structure, where it displaces the water by attaching to the metallic surface, forming a thick and durable anti-corrosive layer. The high penetration potential of lanolin-based anti-corrosive preparations makes them attractive for rust treatment and prevention on automobile parts, especially on old-timers. Some Special anti-corrosive applications and functions

- As a dewatering rust preventative for saltwater-resistant protection of ships and seawater tanks.
- For protection and conservation of ferrous vehicle and vintage car parts such as:
 - crevices of front and car boot lid, wheel house, mudguard, underbody parts and so on;
 - engineering parts like cylinder pistons during storage;
 - chromed parts for better resistance against road salt, seawater and acid rain;
 - for regeneration of bitumen underbody protection.
- For conserving items such as military hardware, conduits, pipelines, machinery, roofing tiles, sheets, plates, tools, steel wire ropes, surfaces of wood and fabric, and so on.

➤ Lubricant applications

Combined with its anti-corrosive potential, lanolin is also a valuable lubricating and conserving material for all types of engineering parts. Other special applications are

- Lubrication grease for engineering parts.
- Metal cutting oil.
- Lubricant for metal processing, e.g. rolling, grinding, pressing and so on.

➤ Leather and textiles

To protect leather from natural degradation processes, it must be treated by chemical tanning agents to create a durable product from an organic source. Lanolin is a widely used ingredient for fat liquors, that are applied after tanning to soften leathers.

Lanolin is used in products for:

- treating and processing tanned leather
- weathered leather, as a nourishing oil and emollient
- shoe polishes, as an emulsifier and gloss enhancer
- Textiles, as an emollient that gives a soft finish.

➤ Other Industrial uses:

- In paints, spraying varnishes and inks:
 - as a dispersing agent for homogeneous covering properties
 - preventing aggregation and precipitation of pigments to improve handling of paints
 - as an agent to decrease the drying time
 - as a penetration inhibitor for inks
 - to give and control fluidity.
- In polishing waxes and abrasives.
- As a paper conditioner:
 - enhancing paper softness
 - imparting steam resistance for wrappings of surgical instruments that have to be sterilized.
- In industrial hand-cleaner creams and lotions as a superfatting agent to minimise the dehydrating effect of detergents.
- In oil-binding agents, due to the high oil-binding capacity of lanolic acid metal soaps.
- In concrete waterproofing products.
- In numerous other applications, such as aircraft glues, conveyor belt wax, special greases, jointing pastes, vulcanising pastes and so on.

VI. CONCLUDING REMARKS

Detergent oriented wool scouring before dyeing is one of the pollution causing process in the mills contributing to the maximum of the total BOD, COD and TDS load in effluent. Lanolin is extracted by scouring the wool fibre in hot water with detergent and other auxiliary chemicals to remove dirt, wool grease (crude

lanolin), suint (sweat salts), and anything else stuck to the wool. The wool grease is continuously removed during this washing process by centrifugal separators, which concentrate it into a wax-like substance melting at approximately 38°C. Separation of wool grease from scouring liquor causes reduction in effluent load contributing less problem areas for waste-water treatment and discharging of wastewater into surface water and also useful to produce value added products (Ex: Lanolin) that are used as a Personal Care like- cosmetics and toiletries, medical application like- ointment bases, burns dressings and wound sprays and in different industrial applications like- Anti-corrosive effect on ferrous metals, Lubrication, Leather and textile industry, Paint and spray vanishes. Lanolin is not a significant allergen. As the incidence of allergies is less than approximately 6 per million then lanolin is less of an allergen than fish, eggs, strawberries, etc. Super-refined lanolin is available recording even lower incidence of allergies which is ideal for the use in hypoallergenic cosmetics such as Lanocreme [13]. Apart from the above mentioned applications, there is lot of scope to produce different value added products by using various derivatives of wool grease. So, future research should be focus on the technical textile application and hygienic and hypoallergenic applications in medical textiles and other industrial applications.

VII. ACKNOWLEDGEMENT

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