بسم الله الرحمن الرحيم

**Sudan University of Science and Technology**

**Faculty of Graduate Studies**

# Manufacturing and Application of Fatliquor from Castor Oil for Upper Leathers

# تصنيع وتطبيق زيت التشحيم من زيت الخروع لجلود وجه

A thesis
Submitted in fulfillment for the requirements of PhD degree in leather engineering

By

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# Dedication

This thesis is dedicated to:
The sake of Allah,
My great teacher and messenger, Mohammed (May Allah bless and grant him),
who taught us the purpose of life,
To the spirit of my father God's mercy on him,
To my mother,
To my wife,
To my family, my sister, my wife and so on.
My friends who encourage and support me,
All the people in my life who touch my heart,
I dedicate this research.

# Acknowledgements

The researcher gives his thanks and appreciation to the supervisor Prof.G.A.Gasmelseed and I.H.Elamin for their help. Thanks are also due to the technical staff of the incubator of the Sudan University of Science and Technology their permit to carry out the experimental work. Also thanks are due to the leather National Research Center, White Nile tannery, for their support and help. Thanks are due to the graduate college of the Sudan University of Science and Technology for giving me this opportunity to do my Ph.D. degree.

# Abstract

Castor oil is classified as semi-drying oil because it oxidizes on exposure to air, giving a gummy film, and can be highly sulphated to make it emulsible in water. In the raw condition it is seldom used in fatliquoring of leathers because crude oil does not emulsify with water and hence the failure of the fatliquor process. Castor seeds were collected, sundried, and crushed. Castor oil was leached in a soxhlet apparatus using hexane as a solvent. An oil content of 28.4%was obtained. Physical and chemical analysis was carried out to obtain crude castor oil. The crude castor oil was converted to fatliquor by sulphation using conc sulphuric acid(98%). The physical and chemical analysis were carried out for the produced fatliquor and compared with the standards. This study is carried, through extraction and characterization of castor oil. Normal hexane was used as a solvent for the extraction process. The characterization analysis revealed that important parameters, include specific gravity, PH, saponification and iodine values for both crude and refined castor oil produced, these were compared the ASTM standard specifications. Sulphated castor oil are added to the leather during re-tannaing process to lubricate leather fibers and to impart softness, flexibility and hand feeling. Fatliquors are mainly emulsifyied mixtures, prepared by addition of phosphate, sulphonate, and sulphite groups etc.Sulphation is one of the methods used to prepare fatliquor emulsion where sulphuric acid is used. In the present study, castor oil is extracted from castor beans. The physicochemical properties of the prepared fatliquor were determined. The fatliquor was applied to leathers in the re-tannaing process and the strength properties of the resultant upper leather were assessed through various physical and chemical tests. The results obtained revealed that castor oil can effectively be used to manufacture fatliquor to be used to lubricate leather in fatliquoring process.

# المستخلص

يصنف زيت الخروع على انه شبه مجفف لأنه يتأكسد عند تعرضه للهواء، ويعطي فيلم غائر، ويمكن سلفنته لجعله قابل امتزاجه في الماء .في حالة الخام نادرا ما تستخدم في تشحيم الجلود لانه لا يمتزج مع الماء، وبالتالي فشل عملية التشحيم .جمعت بذور الخروع، وجففت بالشمس، وسحقت .تم إستخلاص زيت الخروع في جهاز سوكسليت باستخدام الهكسان كمذيب .تم الحصول على محتوى الزيت بنسبة 28.4٪ .تم إجراء التحليل الفيزيائي والكيميائي للحصول على زيت الخروع الخام .تم تحويل زيت الخروع الخام إلى شحم بواسطة الكبريت باستخدام حامض الكبريتيك المضاف (98٪) .تم إجراء التحليل الفيزيائي والكيميائي للمنتج المشحّم ومقارنته بالمعايير.تم إجراء هذه الدراسة من خلال استخراج وتوصيف زيت الخروع .تم استخدام الهكسان العادي كمذيب لعملية الاستخلاص .وكشف تحليل التوصيف أن المقاييس المهمة تشمل قيم الثقل النوعي، الرقم الهايدروجيني، الصابون واليود لكل من زيت الخروع الخام والمكرر المنتج، تمت مقارنة المواصفات القياسية .تم إضافة زيت الخروع المكبرت إلى الجلد اثناء عملية إعادة الدباغة لتليين ألياف الجلد وتحسين النعومة والمرونة وملمس ناعم .التشحيم هو أساسا مخاليط مستحلبة، أعدت بإضافة الفوسفات والسلفونات، ومجموعات الكبريت الخ.السلفنة هي واحدة من الطرق المستخدمة لإعداد مستحلب شحم حيث يتم إستخدام حمض الكبريتيك.في هذه الدراسة، تم استخراج زيت الخروع من حبوب الخروع .تم تحديد الخصائص الفيزيائية والكيميائية للشحم المعد .تم تطبيق الشحم على الجلود في عملية إعادة الدباغة، وتم تقييم خصائص قوة جلود الوجه الناتجة من خلال نتائج الاختبارات الفيزيائية والكيميائية .وأظهرت النتائج التي تم الحصول عليها أن زيت الخروع يمكن أن يستخدم على نحو فعال لتصنيع شحم ليتم استخدامها لتليين الجلود في عملية التشحيم.

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# Introduction

## 1.1 Fatliquoring of leather

Leather, completion time tanning does contain enough lubricant to prevent it from drying in hard. Almost all light leathers need a greater softness and flexibility than is imparted by tannage. This is the process of fatliquoring enter oil on the skin so that the individual is coated fibers.The percentage of oil on very small skin, weight 3-10%. The precise manner in which this small amount is distributed from the oil throughout the skin affects substantially in subsequent finishing processes and nature of skin. Optimal lubrication or fatliquoring significantly affect physical properties of break, stretch, stitch tear, tensile strength, and comfort of leather.Excess oiling will lead to excessive softness and raggy in leather bellies and wings. Under lubrication, or improper penetration, results in hard bony leather that may crack in use. To allow small quantity of oil to the spread in a uniform surface of very large leather fiber that It is necessary to dilute oil. Although this could be done with a true solvent such as benzene, it is cheaper, safer and more convenient to use the method of emulsification. In an emulsion with water, the oil is dispersed in microscopically small droplets, giving it a white, milky appearance. It is important that the oil drops in water should remain as an emulsion until they penetrate the leather, and should not separate out as large drops or as a layer of oil, which could not penetrate the leather fibre and would only give a greasy surface layer (Leather Technician's Handbook J. H. Sharphouse, 1971).

The properties of the finished leather can be varied by controlling the degree to which the emulsion penetrates the leather before it "breaks" depositing the oil on the fibers. By such a technique, in the case of chrome-tanned leather, it is possible to concentrate the bulk of the fatliquor in the surface layers, leaving the middle containing relatively little oil. This yields leather which is soft but resilient, with a tight break. In contrast if the fatliquor is allowed to penetrate uniformly, the leather will be soft and stretchy, with any natural grain looseness accentuated. The commonest material used as a surfactant is soap. However in the presence of hard water, calcium or mineral salts or acid, the hydrophilic nature of the soap is reduced and it loses its surfactant powers. Most leather is acid; sulphated or sulphited, alcohols or oils have much better resistance to these conditions and thus much better wetting action, and emulsions formed with their aid are much more stable to this condition (Leather Technician's Handbook J. H. Sharphouse, 1971).

They are all classed as colloids, anionic or cationic surfactans, depending on the charge of the ionic group carried. Anionic surfactants are more effective at high pH and on anionic materials, eg.vegetable tanned leather.Cationic surfactants are more effective at lower pH and on cationic materials,eg. Chrome leather. Non Ionic surfactants in which the hydrophilic group does not ionize(consists of several hydroxyl groups) are used as auxiliaries in parafin degreasing, as wetting agents, and to stabilize fatliquors to obtain emulsion penetration into the leather(Leather Technician's Handbook J. H. Sharphouse,1971).

Location of the oil:  If we consider a cross section of the hide upon bending, we see that on the outside of the bend the fibers must stretch, and on the inside of the bend must compress. In the center of the skin there is very little motion of the fibers over one another during bending. Therefore both the grain and the flesh surfaces must be lubricated (to prevent break or grain wrinkle), but less lubrication is necessary in the center (Leather Technician's Handbook J. H. Sharphouse, 1971).

Contrary to popular belief, the primary function of fatliquoring is not to soften the leather: this is only the secondary function. The primary function of fatliquoring is to prevent the fibre structure resticking during drying. As the leather dries, the interfibrillary water is removed, allowing elements of the fibre structure to come close together, which consequently allows interactions to occur. In the limit, these interactions become strong, because they are created by the Maillard reaction. Therefore, it is essential for leather quality to prevent this happening.Figure 1.1 shows the structure of a wet blue fibre.1 the photomicrograph is the result of cryo-scanning electron microscopy: the sample is snap frozen in a slush of liquid and solid nitrogen at about –200 1C, then the temperature is allowed to rise a few degrees, so that the water can sublime away under vacuum to leave intact structure. There are several aspects of the photomicrograph to note:

1. Collagenic materials are at their softest when they are soaking wet.

2. The photomicrograph is a representation of wet leather, i.e. no artifacts of drying are present.

3. The fine structure elements are fibril bundles: they indicate a high degree of opening up and the important level of the higher of collagen structure at which opening up occurs is at the level of the fibril bundles.

4. Taking points 1 and 3 together, the sites where lubrication is required are the fibril bundles: this implies that the conditions under which the lubricant penetrates the fibre structure are important.

**Figure 1.1** Cryo-scanning electron photomicrograph of wet blue leather. (A. D. Covington et al, 1993)

Figure 1.2 shows a wet blue fibril bundle at higher resolution there are two aspects of this photomicrograph to note:

1. The structural elements are fibrils;
2. At the surface, where the sample is wettest, the fibrils are separated, but where evaporation of water can begin to take place it can be seen that the fibrils are much closer together.

The opening up processes apply also to the fibril bundles themselves, although it is probable, because of the close association of the fibrils, that the physical properties are derived from the ability of the fibril bundles to distort and slip when stress is applied. Those properties are the elements of handle, including softness, and strength. Handle is a complex concept, because it relates to the way the leather feels when it is manipulated: it is an algorithm that combines density, softness, compressibility, stiffness, smoothness, springiness and stretchiness, easily solved in the brain of the experienced tanner, but much less easy to quantify objectively.

Strength is the ability of a material to resist breaking or tearing stress: in the case of leather, it is the ability of the material to dissipate stress over its volume by movement of the fibre structure. To be able to do this, two criteria must be met

1. The fibre structure must not be stuck together by the adhesions created during drying.
2. The fibre structure must be lubricated to allow the elements to slide over one another.

**Figure 1.2** Cryo-scanning electron photomicrograph of a wet blue fibril bundle.( A. D. Covington et al,1993)

It is the purpose of the fatliquoring step to satisfy those criteria: it prevents fibre sticking during drying by providing an oil surface to the fibre structure, which then gives it the required lubrication. The effectiveness of the process step then depends on the degree to which the lubricant penetrates down the hierarchy of structure and its ability to allow contacting surfaces to slide. The photo micro graph in Figure 1.3 illustrates this, showing the effect of drying without lubricating; whereby the well opened up fibre structure is stuck together at the level of the fibril bundles. Typically, the leather industry employs partially sulfated or sulfited oils, which might be animal vegetable, synthetic or, less commonly, mineral. Most commonly, the neutral oil is in the form of triglyceride (see below). Here, these-called sulfo fraction is the emulsifying agent, keeping the neutral oil suspended in solution and thereby transporting it into the leather: it is the neutral oil that is the lubricant (see below).Oil in water emulsions are created by the formation of particles, consisting of a small drop of oil, surrounded by an emulsifier/detergent/surfactant/ten side.

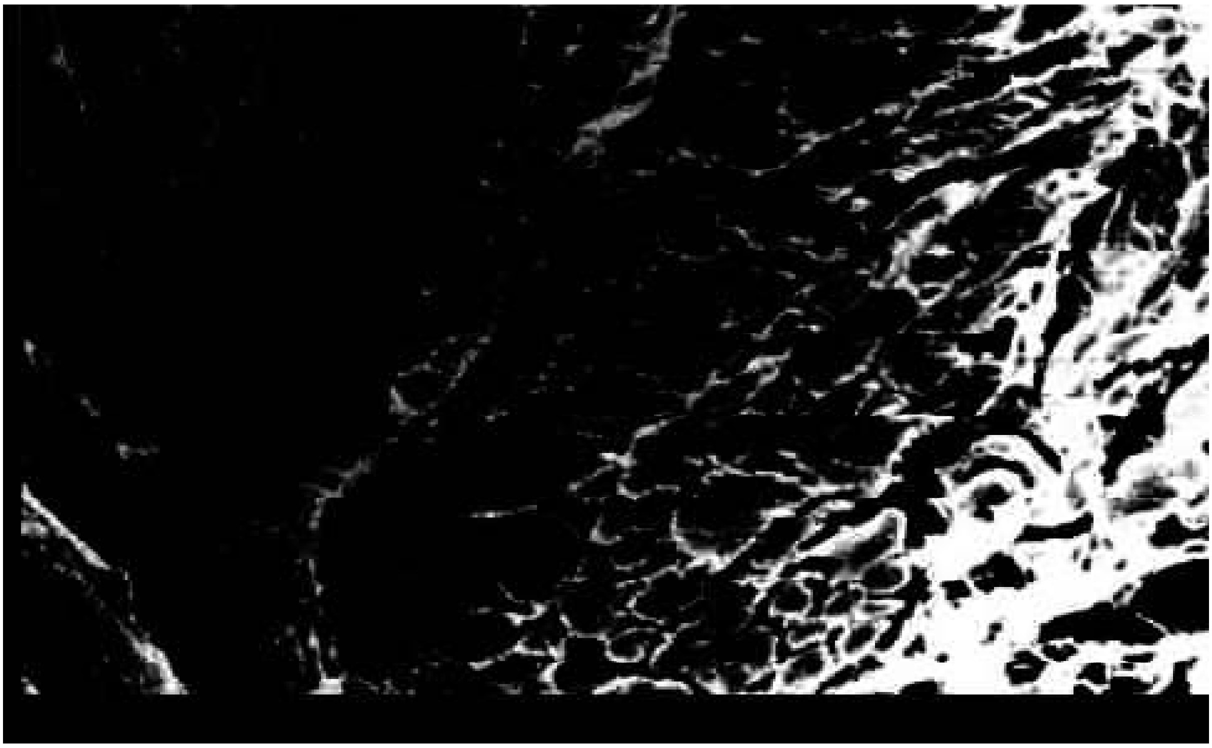


**Figure 1.3** Chrome tanned leather: well opened up in the beamhouse, but dried without lubricant after tanning. (A. D. Covington et al, 1993)

**Figure 1.4** Model of an anionic oil-in-water emulsion particle.( A. D. Covington and K. T. W. Alexander,etal,1993)

The emulsifying agent has a hydrophobic part, which is dissolved in the oil, and a charged, hydrophilic part that interacts with the solvent (water), to keep the particle suspended (Figure 1.4).The emulsion particles are prevented from coagulating or coalescing because they are held apart by the repulsing effect of the high charges on the surface. Any chemical reaction that reduces the charge on the particle surface will allow them to come together, allowing the neutral oil particles to coalesce. High temperature can drive the particles together, breaking the emulsion. An important mechanism to make this happen, which is exploited in leather making, is the ionic interaction between the sulfate/sulfonate group and the protein.

## 1.2 Problem Statement:

Sudan is much endow with vegetables oils which may be treated for fatliquoring of leather, but till now the sudanese tanneries imported oils from abroad, with difficulties in hard currency. The aim of this study is to process fatliquors in order to make it available and reduce the high cost in hard currency. It is hoped that the fatliquor will replace the imported oils and hopefully to be exported.

## 1.3 Oils, Fats and Waxes

Fats and oils predominantly are triesters (triacylglycerols (TAG), triglycerides) of glyecrol and aliphatic fatty acids containing up to 22 carbon atoms. Waxes are esters of long-chain fatty acids, usually containing 24–28 carbon atoms, with long-chain primary alcohols (16–36 carbon atoms) or with alcohols of the steroid group.1Fats and oils are members of a broader group of chemical substances called lipids, which has been classified by the National Research Council into nonpolar lipids including esters of fatty acids (triacylglycerols and cholesteryl esters) that are virtually insoluble in water, but soluble in most organic solvents and enter metabolic pathways only after hydrolysis; and polaroramphipathic lipids including fatty acids, cholesterol, sphin go lipids, and glycerol phosphor lipids (mainly lecithins)( Kent, J,2003).

## 1.4 Mineral oils and waxes

Simplest type is mineral oil, obtained from crude oil from oil wells. They are mixtures of many substances which are separated by distillation. They are relatively cheap and chemically stable and are not affected by mould or bacteria. Can be obtained in pale color. Mineral oils do not mix with water therefore give waterproof properties and can be obtained at any viscosity. Despite the advantages they have only limited use in leather manufacture. Relative to other oils:

a) they are more difficult to incorporate thoroughly without giving a slightly oily or waterproof surface, which is a disadvantage for many leathers which are to be dyed.

b) They have a poor “feeding action”, and used alone they give leathers which feel thin or empty but may be quite flexible.

c) If the resultant leather is heated, the oil may migrate to the surface, which becomes oily or discolored.

These oils do not appear to be as firmly held by the leather fibers as other oils; they are saturated hydrocarbons (unsaponifiable).

Paraffin wax (mp 35-36 C), Montan wax (mp 76-84 C), Ceresine wax (mp 60-85 C).

## 1.5 Natural oils and fats:

Most of the oils and fats in animals, fish and plants are fatty acid glycerides. When boiled with caustic soda, they decompose to give soap and glycerin (saponification). By adding acid to the soap the free acid is formed. These fatty acids are water insoluble and range from very fluid oily liquids to greasy pastes and hard waxy materials. The property of the natural oil is largely governed by which of these fatty acids are combined with the glycerin.

All these glycerides can be split into glycerin and free fatty acid (rancidity) by acids and by action of enzymes (produced by moulds). It may happen to the oil or fat in the leather and if the solid type fatty acids are liberated they may crystallize on the surface of the leather spoiling the appearance of the leather giving a whitish dusty appearance known as “fat spue”. Another trouble due to rancidity of the oil is that free fatty acids form compounds with chromium, alum or zirconium salts used in tanning, which make the leather water-repellent and difficult to wet back uniformly for dyeing or finishing purposes. Fatty acids may be classified according to their chemical reactivity that is their degree of unsaturation. Saturated fatty acids are usually more viscous or solid, do not darken with sunlight, unaffected by damp, warm air, do not combine with sulphur or iodine, difficult to sulphate. Unsaturated fatty acids are more fluid, darken with sunlight, become sticky or gummy on oxidation by air, and readily combine with sulphur or iodine, easily sulphated. Thus highly unsaturated oils may cause trouble on aging of the leather. In the paint trade they are classified as semi-drying(i.e. castor oil) because they become gummy on exposure to air, and drying oils( i.e. linseed oil) which on exposure "dry" to a hard, non-oily or non-tacky varnish.

Practically all naturally occurring fatty acids have an even number of C atoms. Shorter chain saturated fatty acids C-6,C-8, and C-10 are found in coconut and palm oils, milk fat and other softer oils. C-12, lauric acid, is found in sperm oil. Saturated fatty acids of C-16 and C-18 are common to animal fats and many vegetable oils.
The C-24 and C-25 category are found in waxes.ie.carnauba wax and bees wax. The unsaturated fatty acids, primarily of C-18 type are quite common in animal and vegetable oils. Fatty acids with more than 1 double bond are classified as drying oils such as linseed, cottonseed oils. Some contain OH groups such as lanopalmic (C-16 hydroxy, saturated) found in wool fat and ricinoleic (C-18 hydroxy, unsaturated) found in castor oil. Both wool fat (lanolin) or wool grease and castor oil are common fatliquoring materials when sulfated. ( Drevon CA, Nenseter MS, Brude IRet al,1995).

## 1.6 Castor-oil (Ricinuscommunis) plant:

Castor oil is a vegetable oil obtained by pressing the seeds of the castor oil plant (Ricinuscommunis) (Thomas,A,2000). The common name "castor oil", from which the plant gets its name, probably comes from its use as a replacement for castoreum, a perfume base made from the dried perineal glands of the beaver (castor in Latin)( Casselman, W.G,2014). Castor oil is a colorless to very pale yellow liquid with a distinct taste and odor once first ingested. Its boiling point is 313 °C (595 °F) and its density is 961 $kg/m^3$. It is a triglyceride in which approximately 90 percent of fatty acid chains are ricinoleate. Oleate and linoleates are the other significant components (Aldrich Handbook of Fine Chemicals and

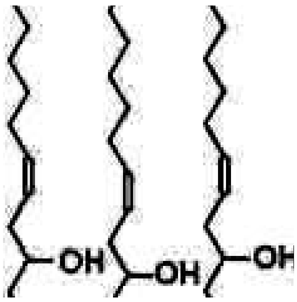
Laboratory Equipment. Sigma-Aldrich, 2003).Castor oil and its derivatives are used in the manufacturing of soaps,fatliquoring of leather, lubricants, hydraulic and brake fluids, paints, dyes, coatings, inks, cold resistant plastics, waxes and polishes, nylon, pharmaceuticals and perfumes (Mutlu, H. and M.A.R. Meier,2010).

**Fig(1-5):**Ricinuscommunis(Allan G, Williams A, Rabinowicz PD,etal,2008)

Castor beans have been found in ancient Egyptian tombs dating back to 4000 b.c. According to the Ebers Papyrus, an Egyptian medical text from 1500 b.c., Egyptian doctors used castor oil to protect the eyes from irritation. The oil from the bean was used thousands of years ago in facial oils and in wick lamps for lighting. Castor oil has been used medicinally in the United States since the days of the pioneers. Traveling medicine men in the late 1800s peddled castor oil, often mixed with as much as 40% alcohol, as a heroic cure for everything from constipation to heartburn**.** It was also used to induce labor. At the present time, castor oil is used internally as a laxative and externally as a castor oil pack or poultice. The castor plant, whose botanical name is Ricinuscommunis, is native to the Ethiopian region of east Africa. It now grows in tropical and warm temperate regions throughout the world and is becoming an abundant weed in the southwestern United States. Castor plants grow along stream banks, river beds, bottom lands, and in almost any warm area where the soil is well drained and with sufficient nutrients and moisture to sustain growth. They are annuals that can grow 6–15 ft (1.8–5 m) tall in one season with full sunlight, heat, and moisture. The tropical leaves, with five to nine pointed, finger-like lobes, may be 4–30 in (10–76 cm) across. Flowers occur on the plant (which is monoecism, meaning that there are separate male and female flowers on the same individual), during most of the year in dense terminal clusters, with female flowers just above the male flowers. Each female flower consists of a spiny ovary, which develops into the fruit or seed capsule, and a bright red structure with feathery branches (stigma lobes) to receive pollen from the male flowers. Each male flower consists of a cluster of many stamens that shed pollen that is distributed by wind. The spiny seed pod or capsule is composed of three sections, or carpels, that split apart at maturity. Each carpel contains a single seed. As the carpel dries and splits open, the seed is ejected, often with considerable force. The seeds are slightly larger than pinto beans and are covered with intricate mottled designs, none of which have exactly the same pattern due to genetic variations. At one end of the seed is a small spongy structure called the caruncle, which aidsin the absorption of water when the seeds are planted. The name "castor" was given to the plant by English traders who confused its oil with the oil of another shrub, Vitexagnus—Castus, which the Spanish and Portuguese in Jamaica called agno-casto. The scientific name of the plant was given by the eighteenth-century Swedish naturalist Carolus Linnaeus. Ricinus is the Latin word for tick; apparently Linnaeus thought the castor bean looked like a tick, especially a tick in engorged with blood, with the caruncle of the bean resembling the tick's head. Communis means "common" in Latin. Castor plants were already commonly naturalized in many parts of the world by the eighteenth century. There are several cultivated varieties of the castor plant, all of which have striking foliage colorations. The castor plant grows rapidly with little care and produces lush tropical foliage. Its use as a cultivated plant should be discouraged because its seeds or beans are extremely poisonous. Children should be taught to recognize and avoid the plant and its seeds, especially in the southwestern United States where it grows wild near residential areas. Flower heads can be snipped off of castor plants as a protective measure. The active poison in the castor bean is ricin, a deadly water-soluble protein called a lectin. The ricin is left in the meal or cake after the oil is extracted from the bean, so castor oil does not contain any of the poison. The seed is only toxic if the outer shell is broken or chewed. Humans and horses are most susceptible to ricin, although all pets and livestock should be kept away from the castor seed. It has been estimated that gram for gram, ricin is 6,000 times more deadly than cyanide and 12,000 times more deadly than rattlesnake venom. A dose of only 70 grams or one two-millionth of an ounce (roughly equivalent to the weight of a single grain of table salt) is enough to kill a 160-pound person. Even small particles in open sores or in the eyes may be fatal. As few as four ingested seeds can kill an adult human. Lesser amounts may result in vomiting, severe abdominal pain, diarrhea, increased heart rate, profuse sweating, and convulsions. Signs of toxicity occur about 18–24 hours after ingestion. Ricin seems to cause clumping (agglutination) and breakdown (hemolysis) of red blood cells, hemorrhaging in the digestive tract, and damage to the liver and kidneys. Ricin has attracted considerable attention as of early 2003 because of its association with terrorist groups. Although ricin cannot easily be used against large groups of people, it has been used to assassinate individuals by injection. The Centers for Disease Control and Prevention (CDC) considers ricin a B-list bioterrorism agent, meaning that it is relatively easy to make and is considered a moderate threat to life. On the positive side, ricin is being investigated as a tool for cancer treatment. A promising use is the production of an immunotoxin in which the protein ricin is joined to monoclonal antibodies. The ricin-antibody conjugate, which is produced in a test tube, should theoretically travel directly to the site of a tumor, where the ricin can destroy the tumor cells without damaging other cells in the patient (Layne, M,2002).

### 1.6.1Composition of castor oil

Castor oil is a vegetable oil obtained by pressing the seeds of the Castor plant (Ricinuscommunis) it is a triglyceride in which approximately 90 percent of fatty acid chains are ricinoleate.Oleate and linoleates are the other significant components.



**Fig(1.6):**Chemical Structure of castor oil(Allan G, Williams A, Rabinowicz PD,etal,2008)

Castor oil is famous as a source of ricin oleic acid, a monounsaturated, and 18-carbon fatty acid. Among fatty acids, ricin oleic acid is unusual in that it has a hydroxyl functional group on the 12th carbon. This functional group causes ricin oleic acid (and castor oil) to be more polar than most fats (Seed Oil Prices, 2008).

### 1.6.2 Toxicity:

The seeds contain 2.8–3% toxic substances, 2.5–20 seed killing a man, 4 a rabbit, 5 a sheep, 6 an ox, 6 a horse, 7 a pig, 11 a dog, but 80 for cocks and ducks. The principal toxin is the albumin, ricin. However, it produces antigenic or immunizing activity, producing in small doses an antitoxin analogous to that produced against bacteria (Devendra, C. and Raghavan, etal, 1978).

## 1.7Turkey red oil

Turkey red oil, also called sulphonated (or sulfated) castor oil, is made by adding sulfuric acid to vegetable oils, most notably castor oil. It was the first synthetic detergent after ordinary soap. It is used in formulating lubricants, softeners, and dyeing assistants (Turkey Red Oil – A defoaming & wetting agent,2014).

## 1.8 Main Objectives:

**1-** Production of fatliquor from castor oil.

**2-** Application of the expermental fatliquor for manufacture of upper leather.

**3-** Determination of the physical and chemical properties of the experimental leather and compare with the standard.

# Chapter Two

# Literature Review

# Chapter Two

## Literature Review

### 2.1 Leather

Leather contains from less than 5% to more than 25% of oil resources by type. Oil is usually introduced while the leather is in the wet state but some oils and waxes are used on dry leather for waterproofing and finishing effects. Animal and fish oils are used extensively both in the raw state and treated to 'make miscible them with water. Oils of vegetable origin have a more limited use.( Newbury, E.W,1940,p43)

### 2.2 Castor Oil

The oil is obtained from extracting or expressing the seed of a plant which has the botanical name Ricinuscommunis. The oil is not only a naturally-occurring resource; it is inexpensive and environmentally friendly. Castor oil is viscous, yellow, non-volatile and non-drying oil with a bland taste and is sometimes used as a purgative. It has slightest characteristic odor while the crude oil tastes slightly acrid with a nauseating after-taste. Relative to other given vegetable oils, it has a good shelf life and it does not turn rancid unless subjected to excessive heat. India is the world's largest exporter of castor oil. (Saxena RC, 1991)

The extraction of oil from castor seed is by one or a combination of mechanical pressing and solvent extraction. In this process of mechanical pressing, the seeds are crushed and then adjusted to low moisture content by warming in a steam-jacketed vessel. Thereafter, the crushable seeds are loaded into hydraulic presses and they are pressed by mechanical means to extract oil. The oil coming from mechanical pressing has light colour and low free fatty acids. However, mechanical pressing will only remove about 45% of the oil present and the remaining oil in the cake can be recovered only by solvent extraction. In the solvent extraction method, the crushed seeds are extracted with a solvent in a soxhlet extractor or commercial extractor. Solvents generally used for extraction include heptane, hexane and petroleum ethers. (Saxena RC, 1991).

As in other vegetable oils, it is usual to refine the crude oil obtained from either mechanical pressing or solvent extraction. The main aim of this refining is to remove impurities (e.g., colloidal matter, free fatty acid, and coloring matter) and other undesirable constituents, thus making the oil more resistant to deterioration during storage. Castor oil, like all other vegetable oils, has different physical and chemical properties that vary with the method of extraction. Cold-pressed castor oil has low an acid value, low iodine value and a little higher saponification value than the solvent-extracted oil, and it is little lighter in colour. (Saxena RC, 1991)

Castor oil-based synthetic detergents are very less prone to foaming and the disposal of the detergent is hastened since microbiological breakdown is simplified.

Fatty acid composition of this kind of castor oil is:

a) Ricinoleic 90%

b) Linoleic 3-4%

c) Oleic 3-4%

Castor in soaps contributes to fluffy, stable lather, conditioning, moisturizing, quicker trace, softer soap. It is often used to super fat soaps. Castor oil should be used at low percentages to avoid overly soft soaps. Also often used in balms, soaps, shampoos, hair oils, and other thick emulsions for the skin and hair (Saxena RC, 1991).

Castor oil is naturally derived oil with unique characteristics that make it an ideal resource with a wide diversity of commercial applications (ICOA, 1992). The extracted oil is one of the first and most dynamic of the industrial oleo chemicals. The functional reactive sites available in castor oil allow its use as a primary product in many industrial compounds.Among all the vegetable oils, castor oil is the most versatile, because of the unique hydroxy fatty acid structure. Castor oil is obtained from the seed by pressing and solvent extraction. Castor oil is processed by running cleaned seed through decorticating machines which remove the seed coat. The remaining endosperm is pressed to remove the oil. Ricin does not partition into the oil because it is water soluble, and the oil is nontoxic provided that no cross contamination occurred during its production.

### 2.2.1 Botanical Characteristics

Castor is predominantly monoecious, with both male and female flowers born on the same raceme (Moshkin and Perestova, 1986). Usuallythe female (pistillate) flowers are found at the apex of the raceme while themale (staminant) structures are generally below. Castor can cross pollinate but usually self-pollinates and does not suffer inbreeding depression (Atsmon, 1989; Moshkin, 1986). Seed capsules form from the pistillate flowers and typically bear three to four seeds. The capsules can have dense rough spines, be spineless, or have intermediate forms.

Capsules can be green, brown, yellow or red during the growing season, but are brown or gray at maturity. Capsules may dehisce at maturity, which occurs along the middle of each locule's outer wall. Seed yields of castor under irrigation range from 900 to 1000 kg/ha and 300 to 400 kg/haunder dry land conditions (Duke and Wain, 1981).Improved varieties in Brazil and the United States yield from 1300 to 5000 kg/ha. Genetic research has increased oil content, yield and shattering resistance.

### 2.2.2 Castor Genetics

Castor has a diploid chromosome number of 20 and tetra ploid species are rare (Moshkin and Dvoryadkina, 1986). Haploids, with a somatic cell chromosome number of 10 occasionally occur and differ from diploid in leafsize and flower development.

### 2.2.3 Ricin and R. communis Agglutinin

The endosperm tissue of castor seeds contains two proteins which are highly toxic (Lord et al., 1994). R. communis agglutinin (RCA), a 120 kDahemagglutinin lectin, and ricin, a 65 kDa cytotoxic lectin, are lethal toeukaryotic cells. They were first described in castor seeds in the late nineteenth century by H. Stillmark, an Estonian scientist, after aqueous extracts of the seeds caused agglutination of mammalian erythrocytes (Franz, 1988). Ricin has two polypeptide chains, A and B, while the agglutinin protein has four polypeptides, linked by disulfide bonds (Butterworth and Lord, 1983). Two of the agglutinin chains are similar to the A chain and two are similar to the B chain of ricin. The A and B chains of ricin, together, are highly lethal to mammalian cells, while the R.communis agglutinin has limited cellular toxicity, but increases agglutination of red blood cells. Ricin E is a variant of the ricin toxin, with an A chain similar to ricin and a B chain hybrid between the ricin and RCA B chains (Ladin et al., 1987).

### 2.2.4 Ricin a Chain

The A chain of ricin is a ribosome-inactivating protein (Lord et al., 1994). This 32 kDa subunit prevents protein synthesis by irreversibly altering the ribosomal RNA subunits involved in translation. The A chain specifically binds to 28S ribosomal subunits, permanently altering its structure. The ricin A chain cannot enter the cell without the B chain. The A and B chains together are highly toxic and are referred to as heterodimeric toxins.

### 2.2.5 Ricin B Chain

The lectin portion of ricin is the B chain. Ricin's B chain specifically binds glycoproteins and glycolipids on the cell surface terminating Inga lactose or N-acetyl galactos amine (Lord et al., 1994). The B chain binds more strongly to complex galato sides than to simple sugars. The B chain has four disulfide bonds providing the galactose/N-acetylgalac to samine binding activity. The N-terminal and C-terminal halves of the B chain contain 41 homologous pairs of amino acids when the two disulfide bonds in each half are aligned. Subdomains formed by the four disulfide bonds may represent a conserved 40 residue peptide which is repeated four times through gene duplication (Roberts et al., 1985). Up to 108 ricin molecules can bind via the B chain to an individual cell by hydrogen bonds (Robertus, 1991).

The B chain attaches to the eukaryotic cell and the intact toxin enters the cell by receptor mediated endocytosis (Bilge et al., 1994). The B chain protects the A chain from proteolytic activities of lysosomes and catharsis.

The B chain may help the A chain become released from cellular endosomes. Mannose residues on ricin can be bound by cellular mannose receptors and also initiate endocytosis (Montfort et al., 1987).Glvcosylation.

### 2.2.6 Ricinoleic Acid

Castor oil is the only naturally occurring source of ricinoleic acid (Canvin, 1963). Up to 90% of the fatty acid in castor oil is ricinoleic acid.

The ricinoleic acid molecule has a carboxyl group, single point of unsaturation and a hydroxyl group (ICOA, 1992). Thisstructure allows for rapid esterification, hydrogenation and acetylation.Ricinoleic acid is used in coatings, fabrics, high grade lubricants, inks, leather preservation, and synthetic fibers.

### 2.3 Olive Oil

Olive oil is one of the most common base oils used in soap making today. 100% olive oil soap or “Castile” soap has been made for centuries – and today, soap makers of all types usually include at least some olive oil in their blends.

Olives are a type of fruit called a drupe which is basically a type of fleshy fruit that has one hard seed at the center. First, the olives are generally crushed and ground into a paste. Then, the oil is to be separated from the paste by various methods. The first oil that has come from the very first crush is the “virgin” olive oil. The paste that is left behind after the first extraction is called “pomace.” Fatty acid composition of Olive oil:

a) Oleic 63-81%

b) Palmitic 7-14%

c) Linoleic 5-15%

d) Stearic 3-5%

Olive oil contributes to soap hardness, stable lather, slippery feel, conditioning, moisturizing. Olive Oil attracts external moisture to your skin, helping to keep skin soft and supple.

Pomace olive oil contains a larger proportion of unsaponifiable ingredients. This slightly affects its SAP value and imparts a greenish color to the oil and to soaps made with it. Pomace oil is preferred to grade an olive oil for soap making.

## 2.4 Neem Oil

Neem oil is obtained from the seeds of the neem tree. The oil is greenish yellow, non-drying with an acrid and bitter taste, and an unpleasant garlic odor. The oil is extensively used to blend other oils in the making of both laundry and toilet soaps in India. The oil saponifies readily and gives a hard-grained soap with good and very stable lather. When used alone for the making of soap it is very necessary to grain the soap as this helps to remove most of the disagreeable odour and colour. On the other hand, if it is used to make soap with other oils, it is advisable to first make neem oil soap. After the soap has been grained, the other oils are stirred into the soap and the required amount of caustic soda solution added to start the saponification again.Neem oil soap is used for both laundry and antiseptic purposes. Neem oil has been used in the manufacture of natural cosmetics, soap, toothpaste, hair and skin products, emulsions, liquors, ointments and medicinal cosmetics (Chatterjee A, Pakrashi S, 1998). However neem oil can be produced mechanically (hot or cold press) or chemically (solvent extraction) from dried neem seeds. The best quality neem oil with a majority of phytoconstituents intact is obtained through cold press. In cold press the oil is lighter in colour and has a milder odour [50]. Moreover potential residual solvents in chemical extracted oil that may pose health hazards to consumers are eliminated since solvents are not used in the pressing techniques (Ramakrishna G, Prasad NBL, etal, 2004,).

Neem oil is rich in essential fatty acids (EFAs), triglycerides, vitamin E and calcium. Because of its EFAs and vitamin D, neem oil penetrates deep within the skin to heal the minute cracks brought on by severe dryness. Fatty acids present in neem kernel oil are

a) Oleic acid (52.8%),
b) Linoleic acid (2.1%),
c) Palmitic acid (12.6%)
d) Stearic acid (21.4%)
e) Other lower fatty acids (2.3%)
f) Linolenic acid (1%)

Acid value of neem oil is <20.0, Neem also stimulates collagen production, good for aging skin. Vitamin E acts as a free radical scavenger, by hindering the oxidizing processes in the skin. It promotes soft and supple skin, helps in reducing old scars and promotes healing. The neem soap will also be slightly antimicrobial. The neem soap will be acceptable to people suffering from skin diseases such as psoriasis and eczema that are allergic to soaps containing Diethanolamine, Isopropyl alcohol, Butylated toluene and Triclosan additives.Neem Oil contributes to stable lather, conditioning. It is said to have the ability to treat a variety of skin disorders such as dandruff (Saxena RC, 1991).

## 2.5 Coconut Oil

On an industrial scale, the dry process is the traditional method of extracting oil from the coconut. This is done by crushing copra in an expeller, the trade name of the machine patented by V. D. Anderson. The meal may be further treated with solvents to extract residual oil. The dry process involves mechanical extraction of oil in crushers or expellers with copra as feedstock (M. H.Gordon and I.A.Rahman, 1991).

The wet-process feedstock is fresh kernel instead of copra. The extracted oil does not have to be refined, unlike the oil from copra. The co products of oil from the wet process are edible. In addition to oil, other edible co products are recovered from the kernel, namely, coconut flour, protein, carbohydrates, and vitamins. To encourage wider commercial application of this process, the following advantages are emphasized: superior quality of the oil product and the recovery of nutrient co products that would otherwise be lost in copra.

## 2.6 Refining

Refining of crude fats and oils involves a series of steps for the removal of impurities from the glycerides to make the product suitable for human consumption and improve product shelf life. The impurities are generally fatty acids, phosphatides, metal ions, color bodies, oxidation products, solid particles, and volatiles that include objectionable dour. Crude coconut oil is refined by any of the following methods chemical refining (batch or continuous) and physical refining (Hassan, L.G, Osoba.etal, 2007).

### 2.6.1 Physical Refining

Coconut oil refiners have gained interest in the physical refining system as a substitute for chemical refining for the following reasons physical refining has lower oil losses vis-a΄-vis chemical refining pollution problems associated with soap stock acidulation is precluded lower installation cost lower steam, water, and power consumption; and distilled fatty acids are of a higher grade than the acid oil from chemical refining (Chatterjee A, Pakrashi S, 1998).

### 2.7 Coconut oil composition

Coconut oil belongs to unique group of vegetable oils called lauric oils. The most abundant fatty acid in this group is lauric acid, $CH_3(CH_2)_{10}COOH$. Other sources of lauric oils are palm kernel, cohune, and cuphea,value ranging from 7 to 12. The saturated character of the oil imparts a strong resistance to oxidative rancidity. Assessment of the oil by active oxygen method (AOM) yielded results between 30 h and 250 h. Although oxidative stability is reduced in some RBD oils, due to losses in the natural antioxidants of crude coconut oils, the addition of citric acid at the end of deodorization as the oil is cooled to 1000C was effective in regaining considerable oxidative stability in the oil.

**Table 2.1 Mean Molecular weight of fatty acid**

| Fatty Acid | Range Weight (Mean) | % |
|---|---|---|
| Caproic C6 | 0.4~0.6 | 0.5 |
| Caprylic C8 | 6.9~9.4 | 7.8 |
| Capric C10 | 6.2~7.8 | 6.7 |
| Lauric C12 | 45.9~50.3 | 47.5 |
| Myristic C14 | 16.8~19.2 | 18.1 |
| Palmitic C16 | 7.7~9.7 | 8.8 |
| Stearic C18 | 2.3~3.2 | 2.6 |
| Oleic C18:1 | 5.4~7.4 | 6.2 |
| Linoleic C18:2 | 1.3~2.1 | 1.6 |
| C20 | t~0.2 | 0.1 |
| C20:1 | t~0.2 | t |

## 2.8 Emulsion

A mixture of one liquid into another immiscible liquid is known as emulsion. The emulsion is not a stable system. When an emulsion is allowed to stand at rest, it will separate into two layers, the upper layer is an oil phase and the bottom being water phase. When the same system is added with an emulsifying agent, it will increase the stability of the emulsion and the separation will not occur. Stability depends on the type, quantity and quality of emulsifying agent added (A. Patel et al, 2003).

## 2.9 Types of Emulsion

The emulsions are of two types. In first type, the oil is dispersed into water and the emulsion is called oil-in-water (o/w) emulsion. In the other one, water is dispersed into oil and called as water-in-oil (w/o) emulsion. Agent which is dispersed is called dispersed phase and the medium is called continuous phase. In o/w emulsion, oil is the dispersed phase and water is continuous phase and in w/o emulsion, it is vice versa. The change of o/w emulsion into w/o emulsion is called as inversion of phase (J. L. Grossiord, M. Seiller, 1998)

## 2.10 Nature of emulsion

An emulsifier should have hydrophilic (HPL) and hydrophobic (HPO) groups. Both groups should be strong but should not be equal. It should be dispersible in organic solvents or water (J. L. Grossiord, M. Seiller, 1998)

## 2.11 Sulfated Fatliquors

For sulfating, the oil must be unsaturated, with a minimum iodine value of 70 the iodine value is the defined as the number of grams of iodine absorbed by100 grams of oil or fat. Oils that have been used in this regard are castor, neat's-foot, soya, groundnut, and cod. Note, after 1980, sperm whale oil was voluntarily abandoned by the global leather industry (A. D. Covington et al, 1993).

The chemistry of the preparation of sulfated oil is in three steps:

1. Preparation: 10–20% Concentrated sulfuric acid on the weight of the oil is added slowly to the oil, with constant stirring. The temperature of the exothermic reaction must be controlled to $<28^0$C, otherwise the oil can char, causing darkening, and the triglyceride oil may be hydrolysedto release free fatty acids. This latter effect can give rise to the problemof 'spue', when the longer chain carboxylic acids can migrate from the internal structure of the leather to the grain surface, visible as a white efflorescence. Diagnosis is confirmed when the efflorescence melts by the effect of a match. Problems associated by overheating can be catalyzed by the presence of iron salts. An alternative is to use a mixture of sulfuric and phosphoric acids (usually in the ratio 0.8: 1.0). This removes the effect of iron (by reaction with the phosphoric acid); the reaction is less exothermic, so the acid mixture can

be added faster. The process is more expensive and results in a mixture of sulfated and phosphated oils (A. D. Covington et al, 1993)

2. Brine wash: Excess free acid is removed by washing the partially sulfate oil with brine, which also separates the oil fraction from the aqueous fraction.

Brine is used to avoid creating an emulsion, which would happen if water alone were to be used. Alternatively, sodium sulfate, ammonium chloride or sulfate could be used. Some hydrolysis of bound sulfate may take place.

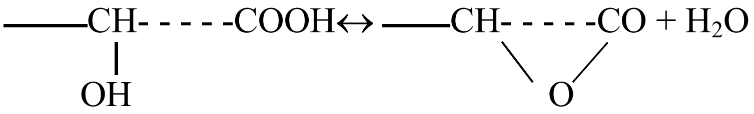
Additionally, there may be lactonisation of adjacent hydroxyl groups with carboxyl groups (Figure 2.1)( A. D. Covington et al,1993).

3. Neutralisation: bound and free acid groups are neutralised with alkali:

$$\text{-OSO}_3\text{H} \rightarrow \text{-OSO}_3^-\text{Na}^+ \text{ or } \text{NH}^+_4$$

$$\text{-COOH (free fatty acid)} \rightarrow \text{-COO}^-\text{Na}^+ \text{ or NH4etc: (water soluble soap)}$$

**Figure 2.1 Neutralisation (T. Waite Proc., Conf, 1986)**



$$\text{——}\underset{\text{OH}}{\underset{|}{\text{CH}}}\text{- - - - -COOH} \leftrightarrow \text{——CH- - - - -CO} + \text{H}_2\text{O}$$

**Figure 2.2 Lactonisation(T. Waite Proc., Conf,1986)**

Bound SO3 may range from 2%, considered a low level, to 3–4%, considered medium level, to 6–8%, considered to be high level: fatliquors may therefore be designated low, medium or high sulfated. Figure 2.2 shows some sulfating reactions. As the level of sulfation increases:

1-Anionic charge increases, hence greater affinity for cationic leather;

2-Lubricating effect decreases, due to the lower concentration of neutral oil;

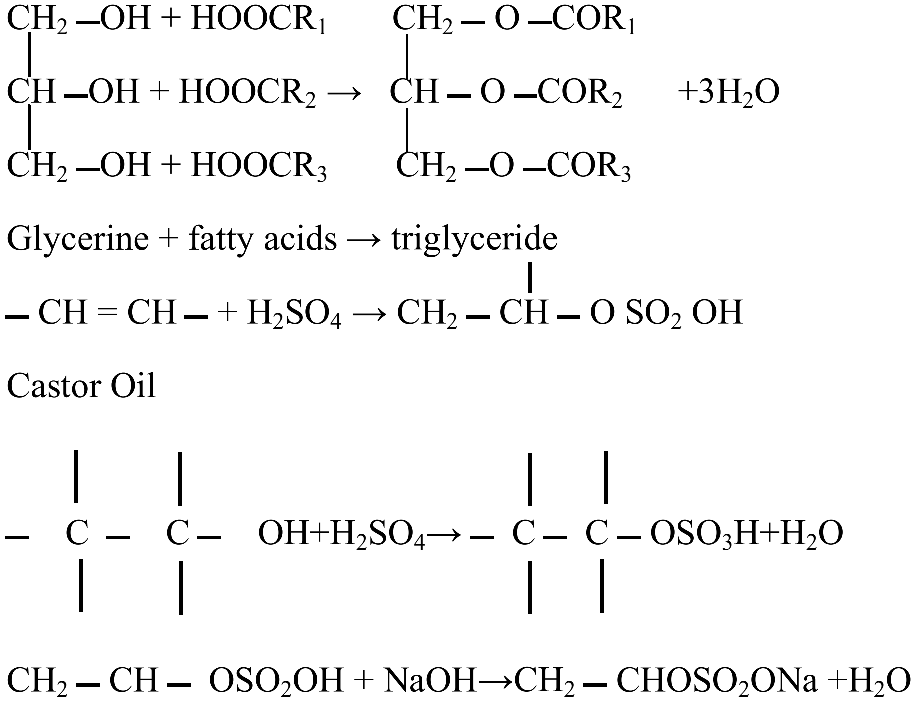
3-Emulsion particle size decreases, ultimately to the point of forming a micro emulsion (<5 nm) or even actually dissolving in water;

4- Stability of the emulsion to coagulation by acid or metal salts increases;

5- At high levels, the oil functions more like a wetting agent than a lubricant, hence the leather becomes more hydrophilic;

6- The leather becomes looser, in terms of break, possibly due to the damaging effect of the sulfate species on collagen;

7- The likelihood of hydrolysing the oil to create free fatty acids increases, thereby creating the possibility of chrome soaps, fatty acid spue, poor wetting back, uneven dyeing and poor finish adhesion (A. D. Covington et al, 1993).



$$\begin{array}{lll} CH_2-OH + HOOCR_1 & CH_2-O-COR_1 & \\ | & | & \\ CH-OH + HOOCR_2 \rightarrow & CH-O-COR_2 & +3H_2O \\ | & | & \\ CH_2-OH + HOOCR_3 & CH_2-O-COR_3 & \end{array}$$

Glycerine + fatty acids → triglyceride

$$-CH=CH- + H_2SO_4 \rightarrow CH_2-\overset{|}{C}H-O\,SO_2\,OH$$

Castor Oil

$$-\underset{|}{\overset{|}{C}}-\underset{|}{\overset{|}{C}}-\;OH + H_2SO_4 \rightarrow -\underset{|}{\overset{|}{C}}-\underset{|}{\overset{|}{C}}-OSO_3H + H_2O$$

$$CH_2-CH-\;OSO_2OH + NaOH \rightarrow CH_2-CHOSO_2ONa + H_2O$$

**Figure 2.3** Reactions of castor oil sulphated (T. Waite Proc, 1986)

## 2.12 Sulfited Oils

The requirement for the oil is unsaturation, as for sulfating. Options included oil, neat's-foot oil, etc. Preparation is conducted in two steps:

1. Sulfitation/oxidation: Using the example of cod oil, air is blown through a mixture of 100 parts of oil and 50 parts of $40^0$ Baume sodium bisulfite solutions, with stirring at 60–80 $^0$C. Alternatively, hydrogen peroxide maybe used instead of air.

2. Brine wash: washing with brine removes excess sodium bisulfite. Note, there is no pH adjustment required, due to the use of bisulfite rather than sulfur dioxide or sulfurous acid (A. D. Covington and K. T. W. Alexander, etal, 1993).

Compared with sulfated oils, sulfited oils exhibit the following properties:

1- No charring or darkening;
2- Higher emulsion stability to acids, hard water salts, metal ions, e.g. Al(III),Cr(III), due to the presence of sulfonate and hydroxyl sulfonate groups and the low level of free fatty acids or soaps, because there is little hydrolysis of triglyceride during synthesis (A. D. , K. T. W,1993).

## 2.13 Soap Fatliquors

Raw oil, e.g. neats foot (100 parts), is emulsified with soft soap, e.g. potassium oleate (30 parts). Note, stearate is likely to cause fatty acid spue. The emulsions have a large particle size, due to their tendency to have low stability to water hardness and acid. The formulation typically has pH~8. At pH<6, the soap is increasingly converted into free fatty acid (Figure 2.1), which does not a ctas an emulsifier, and so the emulsion coagulates. The uses of soap fatliquors are limited, due to the low emulsion stability (A. D., K. T. W, 1993).

They have traditionally been used for surface fatliquoring calfskins for shoe uppers and formaldehyde tanned sheepskin for gloving leather. There is an American practice of improving the emulsion stability by formulating a soap fatliquor with a small amount of low sulfated oil (up to 0.5% $SO_3$ on moisture free basis), together with a stabilizing colloid, e.g. starch or natural gum (A. D., K. T. W,1993)

## 2.14 Cationic Fatliquors

Raw oil is emulsified with a cationic agent, exemplified by the structures in Figure 2.2, where the hydrophilic group is typically straight chain, aliphatic$C_{10}$ to $C_{18}$. The following points apply to such fatliquors:

1- Low affinity for cationic charged leathers, e.g. chrome tanned.
2- High affinity for anionic charged leathers, e.g. vegetable tanned, for lubricating the outer layers.
3- Incompatible with anionic reagents, e.g. anionic dyes, fatliquors, retans.
4- High stability to acid, but unstable to alkali.
5- Good stability to metal ions and salts.
6- May have poor shelf-life.
7- Emulsion stability can be improved by formulating with non-ionic detergents.
8- As a second fatliquor after anionic fatliquor [60].

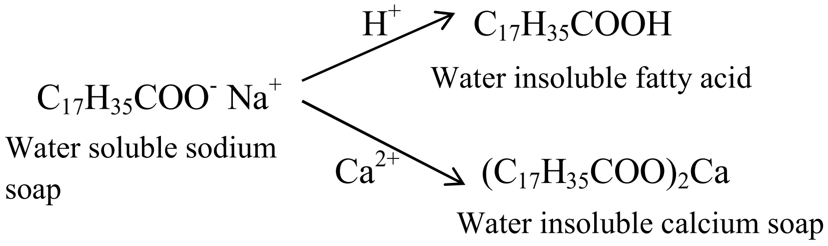


**Figure 2.4** Soap reactions (T. Waite Proc, 1986)

## 2.15 Synthetic Fatliquors

The title “synthetic” is freely used to describe anything from a mineral oil to an etho xylated phosphate ester. Most synthetic based raw materials are available as byproducts of other industries such as from the petrochemical and detergent sector. Medium molecular weight paraffins which are capable of being chlorinated or sulpho chlorinated, oxidized paraffins and paraffin sulphonates being examples. Such products are quite often used in admixtures with naturally occurring oils in order to temper the “dry” handle often associated with synthetic fatliquors.

The advantages they possess over their naturally occurring relatives is that they tend to be almost odourless and depending upon the constitution exhibit little tendency to fatty spues and discolouration.Having said a little about the manufacturing side of fatliquors, it is perhaps noteworthy to say a little about their use (D. BURGESS, .1984).

## 2.16 Application

Today almost a century after the first soap fatliquor emulsions were produced the tanner is faced with a plethora of products from a host of different suppliers.

Technical literature and product information often owes more to the works of Proust than that of Procter. With terms such as soft silky leather of improved tensile strength being liberally scattered about. What is not mentioned is that after fatliquoring, the leather smells like a fishmongers shop on a summers day and darkens on exposure to moon light. The tanner should wherever possible try to obtain basic information as to a fatliquor'smake up and behavior. Performance, limitations or performance characteristics can be assessed in terms of not only softness and fullness but also handle and the effect of a fatliquor on dyestuffs.

As previously mentioned fatliquors enter into the leather in their emulsified form before the emulsion breaks to deposit oil onto and within the fibre weave.

Most fatliquors should exhibit sufficient take up within the first 20—30 minutes running time. Longer floats and lower temperatures will tend to decrease the uptake of fatliquors. Whilst with higher temperatures and lower floats the converse is true.

The type of tannage or retannage will also greatly affect the performance of a given fatliquor. A heavily vegetable retanned leather possessing less affinity for an Anionic fatliquor than a full chrome leather, will tend to exhibit a dry handle and a floating grain surface unless the fatliquor blend is adjusted to have a higher proportion of neutral oil present (D. BURGESS, .2014).

## 2.17 Effect of Dyestuffs

In addition the type, colour and quantity of dye stuffs all have a marked effect upon a fatliquor behavior .Direct dyes of large molecular size and high hydrogen bonding fixation tend to produce firmer leather than a simple monosuiphonated acid dye. In addition the use of large amounts of dyestuff for through dyed leathers has the effect of introducing into the bath high salt concentrations which, depending upon the type offatliquor, will have an adverse effect upon its emulsion stability. Having completed fatliquoring the story is not quite ended. The oil or lubricant present in the leather will displace moisture from the fibre and depending upon the conditions prevailing tends to spread along the fibre and penetrate further. This release of water often being illustrated by the pearling effect seen in fatliquored leather and that freshly fatliquored leather that often appears greasy when removed from the drum is grease free after overnight piling. It is therefore sensible wherever possible to allow the leather to come to equilibrium by piling the rinsed leather overnight on the horse (D. BURGESS, .1984).

## 2.18 Drying

Finally we come to drying; the advent of modern drying systems such as vacuum and high—speed wet toggling has put pressure on fatliquor manufacturers to produce products which show high fibre substantively whilst retaining sufficient lubricant to keep the leather soft. Vacuum drying tends to encourage oil migration and it is perhaps useful to incorporate a highly suiphonated paraffin or sulphonyl chloride type synthetic fatliquor into leather destined for this type of drying.

Wet toggling has the effect of producing leather with a dry handle due to migration of oil away from the grain surface. Leathers intended for such drying methods are therefore best treated with a fatliquoring blend in which a relatively high proportion of neutral oil is present, i.e. a sulphited as opposed to highly sulphated product. Although the looseness factor should be borne in mind when using sulphited products (D. BURGESS, 1984).

## 2.19 Stages in the leather processing

The processing of leather involves three distinct sets of unit operations. These are:

a) Beamhouse or pre-tanning operations
b) Tanning process
c) Post-tanning operations including finishing.

The process flow diagram of raw to finishing operation is given in Figure 2.1.In the beam house operations, the skins and hides received mostly in the wet salted conditions in the tannery are processed to remove unwanted materials such as hair, flesh and other protein us materials and conditioned suitably for the tanning process.

Tanning process is the operation in which the skins and hides are converted into semi-finished leather. In other words, the tanning process imparts resistance to the putrescible skins and hides against bacterial degradation. Apart from this basic function, the tanning materials also impart specific characteristics to the leather, many of which are unique to the nature of the tanning materials used.

In the post-tanning operations, the tanned semi-finished leathers are dyed to give a colour determined by the fashion demand and fatliquored to impart the degree of softness needed in the final leather and retanned to impart the filling, grain tightening and improve the uniformity in substance (Sharphouse, J.H,1979).

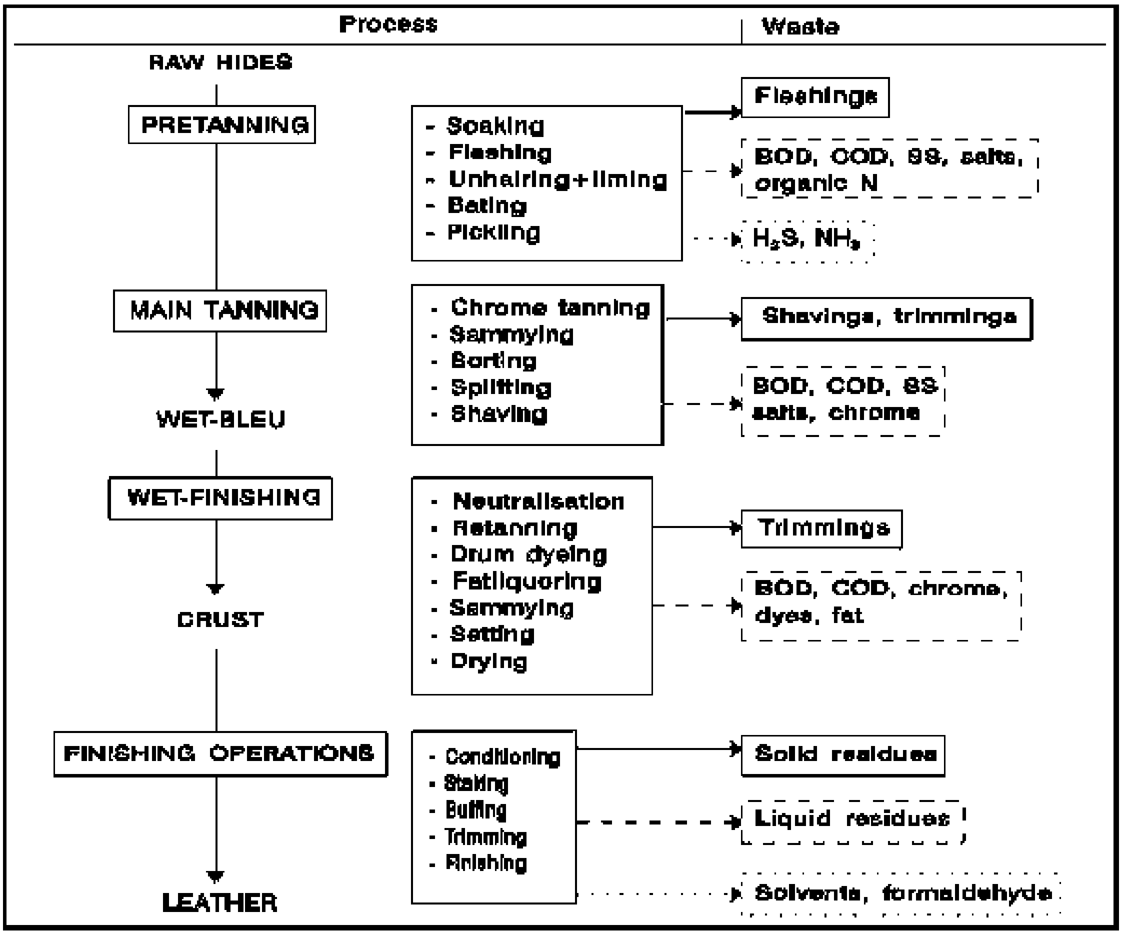


**Fig 2.5:** Flow Chart Leather Processing

### 2.19.1 Soaking

The skins and hides ere received mostly in the wet salted conditions in the Indian tanneries. So, the first operation performed in the tanneries is the soaking operation in which the salt is removed and the good serer hydrated to their original condition (Sharphouse, J.H,1979).

### 2.19.2Liming

a) The major chemical modification of collagen during liming is the hydrolysis of some of the amido groups attached to aspartic and glutamic acid residues(Sharphouse, J.H,1979)

### 2.19.3 Deliming

After washing of the limed pelt to remove lime from the surface, they are delimed with mostly ammonium salts. chlorides or sulphates to bring down the pH to8-8.5.Usually a complete deliming is performed for soft types of leathers where as in the case of heavy. This process is carried out in drums with about 100% water and the subsequent operation, bating is continued in the same bath (Sharphouse, J.H,1979).

### 2.19.4 Bating

The important objective of this unit operation is to purify the pelts by removing the unwanted components consisting of the protein us products, epidermis, short hair and the scud.Inter febrile proteins are also removed during the process to obtain proper grain texture and achieves softness and flexibility in the finished leather. In bating, the proteolytic enzymes based on mostly trypsin are used (Sharphouse, J.H,1979).

### 2.19.5 Pickling

Pickling is carried out to bring down the PH to2.5-3.0 so as to get quicker penetration and more uniform is attribution in chrome tanning. In the case of rapid method of vegetable tanning, partial pickling is done to pH4-.5.

The pelt is treated with a mixture of formic and sulphuric acids, and sodium chloride issued to suppress the acid swelling due to drop in pH and 10% salt concentration is considered necessary to suppress the swelling.80% water based on pelt weight is used in pickling process (Sharphouse, J.H,1979).

### 2.19.6Tanning process

Tanning process is the most important unit operation involved in the leather processing not only because it imparts there is against bacteria degradation and improves the durability but because of the fact that it imparts specific characteristics to the leather like hydro thermal stability, fullness,the charge characteristics and hence the affinity for post-tanning chemicals and auxiliaries (Sharphouse, J.H,1979).

### 2.19.7 Chrome Tanning

80-90% of leathers in the world are tanned by chrome tanning. Chrome tanning uses a solution of chemicals, acids and salts (including chromium sulphate) to dye the hide. It’s a very quick process, taking about a day to produce a piece of tanned leather. First the hide is ‘pickled’ by being left in the acid salt mixture, before being placed into the chromium sulphate. All hides then come out looking light blue (known as ‘wet blue’) and then have a finishing colour applied. Wet Blue is the tanning process where chromes are used to process the leather from raw hide to finished leathers. This process causes the semi-finished leather to look blue-tinted (Sharphouse, J.H,1979).

### 2.19.8 Vegetable Tanning Process

Vegetable tanning is a tanning method that uses natural ingredients such as the bark of chestnut trees to tan the leather. Plant extracts are used for the purpose of tanning in this process. This means the finished product colour is usually warm, rich tones of natural browns. Vegetable tanning is usually done by tanneries that have a rich heritage of tanning hides. Unlike chrome tanning, vegetable tanning can take up to 40 days to produce a piece of dyed leather (Sharphouse, J.H,1979).

### 2.19.9 Fatliquoing Process

Fatliquors have changed considerably over the years, particularly after the introduction of so-called synthetic fatliquors. It has gone through several periods of development from currying and stuffing to soap type fatliquors, and to sulphated, sulphited oils and evolved in recent years into a multiplicity of synthetic lubricants which bear very little resemblance to the soap type fatliquors and stuffing compounds of the past.

Fat liquor normally consists of an emulsifier and a water insoluble oil or fatty matter. The oil or fatty matter serves two main purposes. First, it has to separate the leather fibers to some degree to prevent them sticking together during drying and secondly, it has to act as a friction reducer enabling the fiber to pass against each other without too much resistance or squeak. A further requirement should be to impart the desired feel and gloss on suede or aniline leather, or to prepare the surface for finishing. A useful leather oil should be water insoluble, of reasonably high molecular weight, non-volatile, fairly linear in structure (Heavily branched molecules are unsuitable) and a reasonably viscous fluid or paste. These oils need to be emulsified to reach inside the leather in water medium. For these purpose, there are a wide variety of emulsifier available in the market. They are a means of dispersing the lubricants very finely in water to enable it to penetrate into the leather. Such emulsifier should have the desired property of allowing the emulsion to break at a point predefined by the tanner to give him the required penetration and complete uptake of the applied lubricate. In order to do this, they must be the anionic or cationic. Although non- ionic emulsifiers are used, it is primarily as an adjunct to other systems.

The emulsifiers are responsible for:

1- The electric charge
2- The stability
3- The degree of dispersion of the emulsion
4- The binding power of the respective fatliquors to leather
5- The reaction towards the influence of water.

Properly controlled sulphation process, the properties of sulphated products depends on the following factors:

1- Strength of the sulphuric acid
2- Proportion of the sulphuric acid to oil used

3- Rate of addition of the sulphuric acid

4- Degree of mixing or agitation. (Sharphouse, J.H,1979)

### 2.19.9.1 Fatliquoring of Leather

The oil in water or water in oil emulsion which is used for the lubrication of the tanned fibres of leather is called as fatliquors. The contents of a fatliquor are given below:

### 2.19.9.2 Raw oils

These do not get fixed to the leather fibres but lubricate the fibres.

### 2.19.9.3 Surfactants

This will emulsify the raw oils and carry the inside the leather easily. At the same time it will get fixed to the leather fibres. Though fixed to the leather fibres, the hydrocarbon chains of these surfactants lubricate the leather fibres. Also, prevent the free movements of raw oil molecules inside the leather by their power of adhesion (Sharphouse, J.H,1979).

### 2.19.9.4 Continuous phase

This continuous phase acts as a vehicle for the oil droplets surrounded with emulsifiers. In most of the cases, the continuous phase is water. In some special cases, high boiling point petroleum solvents are also mixed with water and used as continuous phase (Sharphouse, J.H,1979).

### 2.19.9.5 Requirement of fatliquoring

Cryo-scanning electron microscopy photomicrographs of wet leather, dried leather without fatliquoring and dried leather after fatliquoring were given in the following Figures 2.5 and 2.6.

Collagenic materials are at their softest when they are soaking wet. The structural elements of the wet leather are fibrils and indicate a high degree of opening up which requires the lubrication during drying.

The fibre structure must not be stuck together by the adhesions created during drying. It must be lubricated to allow the elements to slide over one another. It is the purpose of fatliquoring step to satisfy the criteria (Sharphouse, J.H,1979)

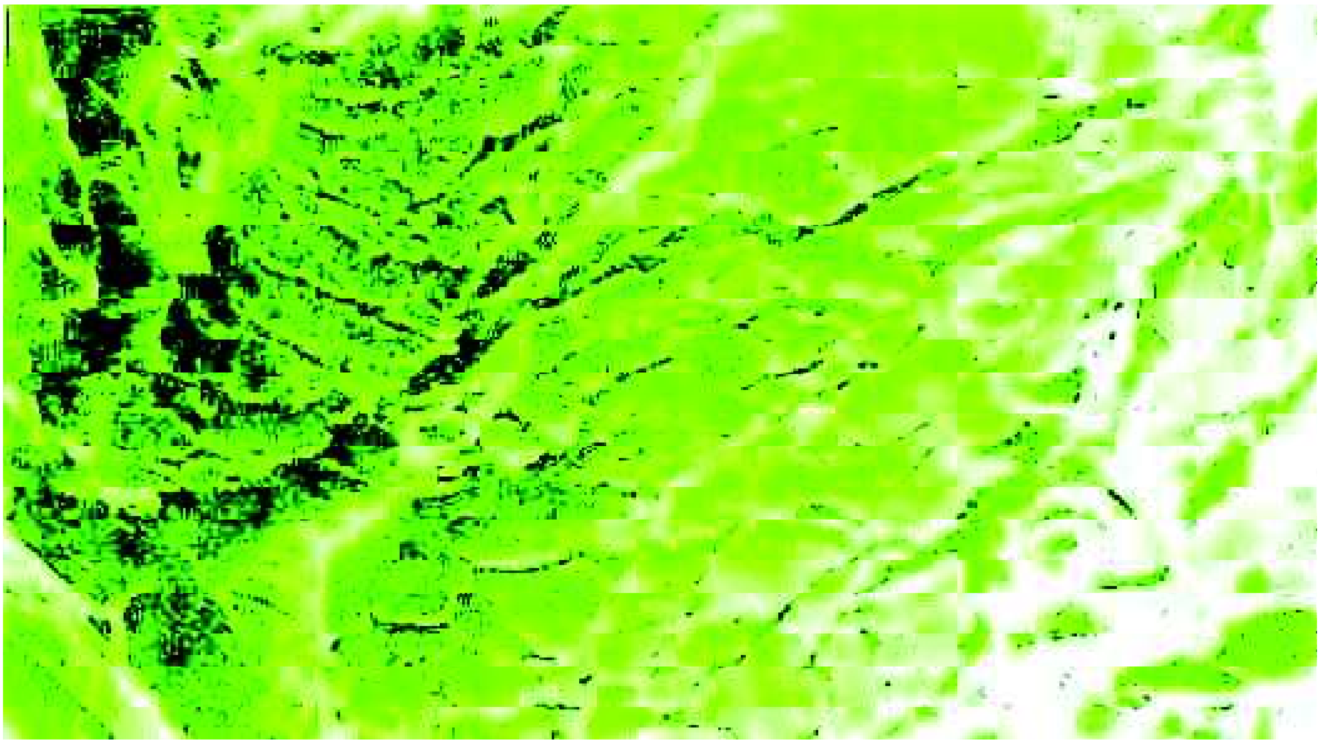


**Figure 2.6** Dried leather without fatliquoring(A. D, etal Covington,1993)

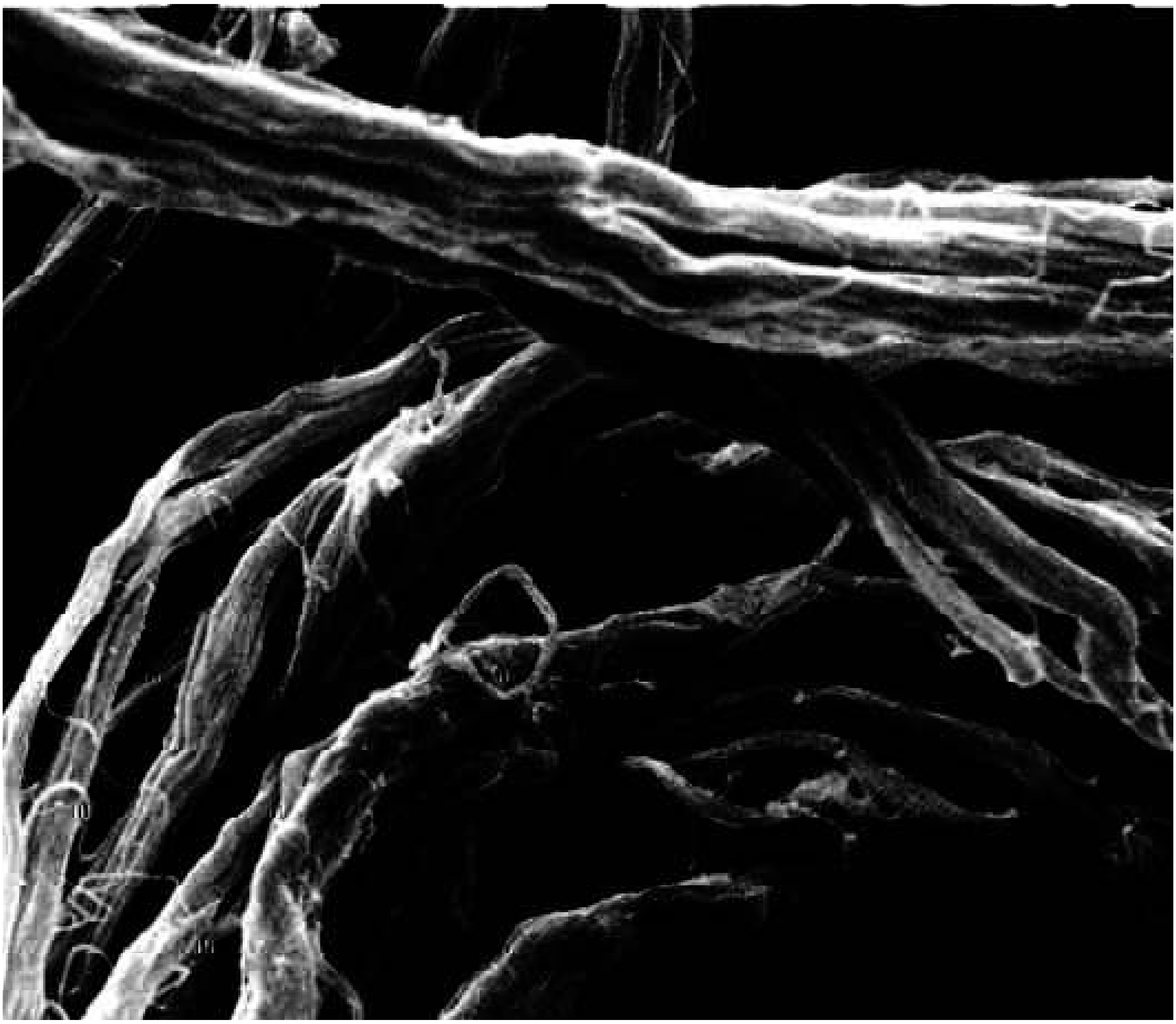


**Figure 2.7** Dried leather after fatliquoring(A. D, etal Covington,1993)

If the leather is dried without fatliquoring, the well opened up fibre structure is stuck together at the level of fibril bundles. To maintain the leather fibrils as in Figure 3.0 and 5.0 after drying also, which is a prime requirement to prevent the fibre structure restocking during drying and also to be soft, fatliquoring step is carried out (Sharphouse, J.H,1979)

### 2.19.9.6 Classification of fatliquors based on the nature

Fatliquors were originated during the twentieth century and classified based on its nature as described below:

### 2.19.9.7 Natural Fatliquors

Vegetable oils like drying oils (linseed oil and hemp oil), semi drying oils (soyabean oil and cottonseed oil), nondrying oils (olive oil and castor oil), vegetable fats (coconut fat and palm kernel fat), animal oils (fish oil and shark liver oil), animal fats (mutton tallow and butter fat) and waxes like vegetable origin (carnauba wax and montan wax), animal origin (beeswax and wool grease) are the examples of this category (Sharphouse, J.H,1979).

### 2.19.9.8 Synthetic Fatliquors

Imitation of natural oils (Neats foot oil and sperm oil) and substituted natural oils (chlorinated oils, silicones, polybutenes and paraffin waxes) belong to this group.

### 2.19.9.9 Classification of fatliquors based on charge

The fatliquors can be classified based on its charge, into anionic (sulphated44, 45,46 -C-O-$SO_3$, and sulphonated47-C-C-$SO_3$), cationic (salts of tertiary amines or quaternary ammonium compounds48), nonionic [oxidecondensates, (-O-O-)] and amphoteric (amino acid type) as per their charge. (Sharphouse, J.H,1979).

### 2.19.9.10 Principles of Fatliquoring

In the production of leather, fatliquoring is usually the last operation in the wet end before drying. This process is generally carried out using oil in water or water in oil type emulsion based fatliquors. It is of decisive important step for the quality and properties of the leather. The fatliquoring process determines the mechanical and physical properties of the leather. The principles of modern fatliquoring methods are based on the following important points:

a. Instead of waiting for long time to develop the opposite charge in the neutralized leather, for the rupture of electrically charged fat droplets inside the leathers, some chemicals having charge opposite to the charge of fat-droplets may be introduced into the leather after fatliquoring. These materials of opposite charge may also have lubricating and filling properties. Such emulsifiers produce stable oil in water emulsion and fix very strongly to the leather through ionic hydrophilic end. It will reduce the migration of lubricating oil by firmly adhering them by the hydrophobic end.

In such cases the attraction between the lubricating oil and the hydrophobic end of the fixed emulsifier becomes so strong that the solvents used for fat determination cannot appreciably extract the oils of the leathers. Thus, the extractable fat content of the leather remains low.

b. Fats and oils of highest lubricating power should only be selected for the preparation of fatliquors, so that small amount can produce sufficient softness to the leather.

c. The emulsifiers may or may not possess any lubricating power but carry the lubricating oil deep into the leather without facing any resistance during penetration.

d. The strengths of both the hydrophilic and hydrophobic ends of the emulsifiers should be as high as possible but among them, the hydrophobic side should be slightly stronger than the ionisable hydrophilic side (Sharphouse, J.H,1979).

### 2.19.9.11 Typical parameters for making a good fatliquor

To prepare a fatliquor, the base can be mixed with a specified quantity of raw oil, emulsifier and a continuous phase. For the preparation, the following parameters are kept constant:

1. Order of addition: Water to oil or oil to water,
2. Ratio of oil to water,

3. Amount of agitation,
4. Temperature of water,
5. Length of time is held prior to running it into the wheel and
6. Water hardness.

### 2.19.9.12 Factors controlling fatliquoring

#### 2.19.9.12.1 Float

If the ratio of fatliquor to water is high, there is a chance of less absorption of the oil. The float in fatliquoring bath should be confined between 80-125% on the shaved weight of the leather (Sharphouse, J.H,1979).

#### 2.19.9.12.2 Nature of oils and fats

The nature of each oil making up the fatliquor (viscosity and drying properties) confers certain distinct properties to the finished leathers (Sharphouse, J.H,1979).

#### 2.19.9.12.3 Temperature

With the increase in temperature of the fatliquoring bath, the extent of fat take-up diminishes (Sharphouse, J.H,1979)

#### 2.19.9.12.4 pH:

Higher acidity causes premature splitting up of fatliquor before it has a chance to penetrate into the leather. This results in undernourished leather of greasy surface.Hence, starting the fatliquor operation at higher pH to get necessary penetration, followed by acidification to a pH of 3.5 – 4.0 to get greater fixation of the oil on the leather (Sharphouse, J.H,1979).

## 2.20 Kinetics of the catalytic esterification of castor oil

The esterification of castor oil with lauric acid was investigated using tetra *n*-butyl titanate (TBT), $SnCl_2 \cdot 2H_2O$ (stannous chloride), $CoCl_2 \cdot 6H_2O$ (cobalt chloride), and $(CH_3COO)_2Zn \cdot 2H_2O$ (zinc acetate dihydrate) as catalysts. Effects of catalyst concentration and reaction temperature on the progress of the reaction were investigated. TBT was the best catalyst for the esterification of castor oil with lauric acid at temperatures lower than 200°C. The reaction was first order with respect to each reactant. The activation energy for the esterification reaction of castor oil with lauric acid using TBT was 26.69 kcal/mol. The rate constants obtained for the esterification of castor oil with decanoic, lauric, palmitic, and stearic acids were nearly the same (15.80, 15.44, 15.06, and 14.67 mL $mol^{-1}$ $min^{-1}$), as were the rate constants obtained for the reaction of castor oil and hydrogenated castor oil (Mangesh G. Kulkarni, Sudhirprakash B,2003)

## 2.21 Preparation and properties of nano-ZnO/modified ydrogenated castor oil composites

Leather products are made from the natural skin collagen fibers. It is vulnerable to the environmental factor such as solar ultraviolet irradiation in the using process. Therefore anti-UV performance is a very important quality, particularly for chrome-free leather. ZnO is a well-known UV absorber commonly used in the cosmetic industry. We have investigated its potential to increase the anti-UV performance of chrome-free leather. Modified hydrogenated castor oil/GPTMS-ZnO (MHCO/ GPTMS-ZnO) composites were prepared using spherical ZnO nanoparticles, hydrogenated castor oil, maleic anhydride and sodium bisulfite. MHCO/GPTMS-ZnO composites have better anti-UV ability and stability. MHCO/GPTMS-ZnO composites were applied to the leather processing. The treated samples were exposed to artificial sunlight. Anti-yellowing tests showed thatMHCO/GPTMS-ZnO composites significantly improved anti-UV performance of leather (Ma, J., Duan, L, 2017).

## 2.23 Preparation and application of castor oil

Castor oil in water emulsions known as fatliquors is used for the lubrication of tanned leather fibres to get softness and also to improve the strength properties. Fatliquoring agents are usually obtained by chemical modification. In this research, fatliquoring agent based on castor oil was prepared using nano-$TiO_2$ as emulsifying agent by mechanical mixing. So castor oil/nano-$TiO_2$ composite fatliquoring agent (CTF) was obtained via Pickering emulsion method. The presence of nano-$TiO_2$ resulted in a stable emulsion. The results of transmission electron microscope showed that the castor oil was surrounded by nano-$TiO_2$. Finally, CTF was applied in the leather fatliquoring process of goatskin garment. Physical and mechanical properties of leather fatliquored by CTF were improved compared with those of leather fatliquored by sulfated castor oil. CTF was easily biodegradable as compared to modified castor oil fatliquoring agent (Lv, Bin and Wang, Hong-Di and Ma,2016)

## 2.24 Castor oil sulfonated

Castor oil sulfonated can directly with $SO_3$ commercially available continue a batch of equipment. Product good color excellent water high lipid solubility and improve stability acid rain, the process of the following advantages. It is more than much faster than this process. It requires the shorter working hours, and is therefore more economical. Can be given a wide variety of products of the different characteristics of high lipid solubility combined content. Since the reaction batch or persist, waste stoichiometric constitute remove any pollution problems (Kremers, F.J,1971).

N- Sulfonated castor oil. The term is sometimes applied to sodium salt this product, oil product is primarily the reaction of sulfuric acid with groups hydroxyl castor oil, but also some secondary reactions. Turkey red oil used as a wetting agent (Gooch, J,2011).

## 2.25 The Formation of Emulsifying Agent by the Partial Neutralization of Sulfated Ricinolic Acid

The possibility of preparing some emulsions by partial neutralization of high molecular weight carboxylic acids has been mentioned in the literature, This technique leads to the formation of emulsions consisting of free carboxylic acid and their salts (e.g sodium salts) in the same time. Based on that, an attempt was made to form an asphaltic emulsion containing an emulsifying agent prepared by the partial neutralization of sulfated ricinolic acid. The emulsifying agent was prepared by the sulfation of ricinolic acid up to 2.3 % (by weight) of $SO_3$ content. Partial neutralization of the resulting sulfated acid was carried out using a concentrated solution of sodium hydroxide to afford 25% neutralization. This allowed a good level of solubility in water. An asphaltic emulsion was prepared from water, toluene and liquid petroleum asphalt in ration of 1: 1: 1 (by weight) in the presence of 2.0 % (by weight) of the emulsifying agent. This emulsion showed high level of stability. For comparison, the partial neutralization of ricinolic acid resulted to no emulsification properties (Imad H. Al Wahaib, 2009).

## 2.26 Synthesis and Properties of Alkoxysilane Castor Oil

The investigates the structural modifications of castor oil a renewable resource, to develop functional organic inorganic hybrid coatings (ShaikAllauddin,et al.,2003). A novel methodology has been developed to introduce hydrolyzable $-Si-OCH_3$groups in the castor oil backbone that has been used subsequently for the development of polyurethane/urea–silica hybrid coatings. The alkoxysilane functional castor oil (ASCO) was characterized by techniques such as $^1H$, $^{13}C$, and $^{29}Si$ nuclear magnetic resonance spectroscopy (NMR), Fourier transform infrared (FTIR) spectroscopy, gel permeation chromatography (GPC), differential scanning calorimetric (DSC), and thermo gravimetric analysis (TGA). The ASCO was further reacted with different ratios of is opho ronedi iso cyanate (IPDI) to get an isocyanate-terminated hybrid polyurethane prepolymer that was cured under atmospheric moisture to get the desired coating films. The glass transition temperatures (Tg) of the hybrid networks were found to be in the range of 29–70 °C, and the water contact angles were in the range of 75°–82°. The Tg and hydrophobic character of the hybrid coating films found to increase with an increasing NCO/OH ratio. The thermo-mechanical, viscoelastic, swelling, morphological, and contact angle properties of these films were evaluated. The alkoxysilane-modified castor oil-based coatings have shown better mechanical and viscoelastic properties in comparison to the control (unmodified castor oil) coatings. This work provides an effective and promising way to prepare hydrolysable silane functional castor oil for high performance hybrid coatings.

## 2.27 Fatliquor preparation from Karanja seed oil

The fatliquor affect the physical properties of skin and make it more flexible, and softer (Ariful Hai Quadery., 2015). fatliquor of seeds oil karanja of sulphation process by adding sodium hydroxide to maintain the pH at 5.0 with conc sulfuric acid. FT-IR sulphated analysis confirmed the product annex of sulphonic acid group. Physical, chemical and biological characteristics fatliquor were satisfactory. The preparation of fatliquor processing application goat skins. Addressing physical and chemical characteristics of goat skins compared with leather processing of fatliquor from castor oil and all the remaining address leather and chemicals in addressing. The goat skins of these two types of fatliquor found leather standard specifications. Even fatliqour karanja of oil extracted from seeds soxlet extraction method is an alternative fatliqour imported.

## 2.28 Synthesis of fatliquor from waste

The fatliquor waste had been compiled from beef fat for use in the leather industry (B C Nyamunda,et al.,2013). The physical properties - Chemical fat before sulphation with sulfuric acid and then with the neutralization of ammonia production fatliquor.The fatliquor chemical and physical analyzes. The degree of sulphation found that 90% of the surface active groups of fatliquor adherence to and form. The fatliquor have been applied in the light of the experiences skin physical hard on leather. The results of the physical tests specific leather consistent with standard specifications. It is noted that the compendium fatliquor can be used as an alternative to the skin.

## 2.29 Preparation of Modified Rapeseed Oil/Organic Montmorillonite Nanocomposite

The Modified montmorillonite myristic acid through intercalation reaction montmorillonite (OMMT) obtained (LBin1,et al.,2013). It also prepared ommt, rapeseed oil,diamine hydrate, and hydrazine aqueous); ethylene, and sodium bi-sulfite working on the preparation of amendment of rapeseed oil (MRO) /ommt nanocomposite (MRO/ OMMT) fatliquoring customer. ommt structures and MRO/ommt achieved flourier currency spectral analysis infrared (IR) - X-ray diffraction (XRD). The stability of MRO/ommt determined by dynamic light analyst (DLC) Emulsifying disc stability tests. MRO/ommt fatliquoring process in the skin. Physical Characteristics and mechanical properties of crust retardant plastics that are reduced after fatliquoring was identified. On the feet of the results of the infrared rays that MRO/ommt successfully. The results indicated that XRD interlayer spacing of MMT of 1.239NM NM to 1.517after amendment myristicacid.skin samples treated with MRO/ommt Excel flame retardancy and mechanical properties on samples of skin MRO axle.

## 2.30 Synthetic fatliquor for low fogging upholstery leather

patent more or de sulfosuccinates of ethoxylated and/or propoxylated fatty alcohol with a number of spies from co more 7 treatment of skin fatliquoring step consists in absorption composition includes 1 to 30% of the weight of said or de sulfosuccinates (Fornara,et al,.2003).

## 2.31 Fatliquors

The fatliquoring tunes used in the treatment of metals tanned leather effective alternatives to oil, which traditionally used to soften the skin (Peratello,et al,.1998). The literature, which include monomers polymerize on site and softening skin break fiber leather to smaller units can move when drought skin. Useful unsaturated lactic acid-glycolic acid monomers is higher choline esters such as lactic acid-glycolic acid, unsaturated fatty monomers such esters of unsaturated fatty alcohol and acrylic acid esters vinyl unsaturated fatty acids. The researcher Peratello, S.D,et said in his paper, Leather fatliquoring agent working the process of using the sulfosuccinic (C12-24 fatty remains) Ester, and tension emulsifier or heat sinks are shipped. The customer is stable in the presence of tanning wine electrolytes (Friese, H.H,1988).

## 2.32 Sulfonated resins

The sulfonated resins useful retanning and fatliquoring agents obtained from condensation sulfonation product (Peratello,et al,.1978) (a) One Mall of volatile hydroxy benzene. Aromatic component, (b) from 0.05 to 1.5 Mall Of Oil-saturated and (c) about 0.4 to 1.0 of the mall by formaldehyde edit configuration. He said al-Otari volatile hydroxy benzene. It may be a component, a lower alkyl phenol at least hydrogen reaction in 2, 4- or 6-The aromatic ring or a combination of them. Non-saturated castor oil or moellon. The liberation by formaldehyde may be configured by formaldehyde or configuration that is capable of producing and formaldehyde had been used for biological decontamination. Formation of formaldehyde or formaldehyde edit formation may be able to produce formaldehyde.

## 2.33 Partial phosphoric esters, preparation thereof and use thereof as leather fatliquoring agents

The leather from fatliquored average absorption of soluble and partially water dispersion polyalkylene phosphoric esters of glycol monoethers of higher Aliphatic compound alcohol fatty acids, in the form of mineral salts or ammonium or alkali with visual after treated preferably mineral (especially from the same absorption medium [Amati, W,.1983]. If you want to be dyed with leather dyes customary skin before or after treatment with fatliquoring agents the invention or before subsequent processing with polyvalent metal cation. Partial phosphoric esters are useful in the form of some of the formulas (Amati, W,1983).

## 2.34 Polymeric retan fatliquor for low fogging upholstery leather

The way to treat skin lower functional fog retan fatliquor include fragmentation amphiphilic selection of stages, with a high degree of freedom from organic solvents consisting of giving precedence to amount to at least a sugar monomer, toluene diisocyanate, are hydrophobic and insoluble small amount of at least one and aquaculture, and plants copolymerizable a sugar monomer [ Hodder, J,et al,.1994]. This method produces leather and the desirable characteristics of the Force, forbearance, in particular reduce fog skin characteristics in particular suitable for use in the vehicle.

## 2.35 Composition and process for the retanning and fatliquoring of leather

To this invention to tracks on retanning and fatliquoring by tanned leather, in the process of the preparation of this literature to the use of the works on the preparation of leather ) "leather" include Fur, leather or hits), as well as a novel type of skin, which can be obtained from the use of the installations.( Komforth, R,.2013)

## 2.36 Preparation of surface-modified silica nanoparticles containing Fatliquor

The surface silica Nanoparticles Could Improve Gene Therapy amended fatliquor includes preparation [Lu Bin,et, 2015]. At the present time, the introduction of silica surface meters amendment fatliquor researchers and technical preparation of fatliquor nanocomposite rarely reported. This invention is modified Under-Secretary -lubricants castor oil at room temperature with stirring the surface ultrasound. Amendment of Nano silica was prepared containing the amendment Nano silica fatliquor. This invention is modified castor oil, consensus, easily, easily integrated into, environmentally friendly leather chemicals after fat crust skin plush soft and some water solvent mechanical resistance and plastics that are reduced have improved significantly reduced (Preparation of surface-modified silica nanoparticles containing Fatliquor,2015).

## 2.37 Innovation of New Cleaner Technology Tannage

The tannage of clean technology using plant oils (Garad) tanning alternatives to aluminum and chrome [Ebtesam A. Hassan,et al,.2014]. 12 pickled treatment of cattle hides pilot drum speed of 8 rpm four paths.Use nilotica Djerad (Acacia) client provides tannage Green (30%) Aluminum retannage in different percentage (1, 2, 3, 4%), followed by 10% locusts. The keystone of the chemical analysis of the skin humidity, ash, fats and pilot implemented leather.

Physical testing, including the thickness of the snap, percentage of elongation so that more women in the reporting interval contraction of temperature. Chemical characteristics of leather in all trials normally. The contraction of temperature for each pilot tracks leather higher than 70 degrees Celsius, tighten over 215 kg/cm$^2$(Ebtesam A. Hassan1, M.T.I.a.S.K,2014)

## 2.38 Application of Acacia nilotica spp nilotica Pods (Garad )Powder as Alternative Vegetable Retanning Material

The retannage is a process used to improve roundness, cereals, leather packets fill using plant oils syntans tannins, various types of resins (A.E. Musa,et al,.. The objectives of the process to fill the inaction of the softer parts of the skin for the production of leather and physical characteristics of more consistent, to allow for the production of unlined cesspits footwear to improve stability chemical to allow quick finishing and delivery to the customer.Nilotica powder Acacia sppnilotica pods (Garad) widely in the Sudan had been evaluated for use in leather retanning presented in this paper. The locusts powder retanning used wet, leather, blue.The effectiveness of the locust retanning powder blue wetlands were compared to the fence leather retanning organoleptic.

The characteristics of the leather, corn, fullness, and transmission cereal grains (ARM) roundness, general appearance, the unification of the retanned dyeing of locusts has evaluated the leather and compared the fence retanned leather.

Locusts resulted in leather retanning with cereals. Dyeing retanned characteristics Jarad, leather, indicating that the best of the fence retanned leather. Also a feature of physical force and shrinking and the temperature (Gasmelseed, A.E.M,2014)

## 2.39 Fatliquoring agent and drying temperature effects on leather properties

Leather, collagen materials structure usually requires Add Clients fatliquoring recently causing instability during heating in the manufacturing processes. The impact of the fatliquoring focus and drying the temperature of the Working Group on the skin of property. Leather and leather products and absorption of the contraction of water, water vapor permeability measured under the specific circumstances. Samples of skin inflammation obtained after the whitewashed tanning chrome commercial use of operations. The samples were fatliquored with 3, 6, 9 and 12% solutions abstract fatliquoring synthetic client, dried in different temperatures. The results of the investigation that has been confirmed by the survey of electronic microscope (SEM) study. They have demonstrated how the focus fatliquoring impact of the Working Group and the freezing temperatures of the leather (Bajza, Z.V.V,etal,2001)

## 2.40 Highly Stable Nonionic Fatliquors Based on Ethoxylated Overused Vegetable Oils

Overused vegetable oils, which are considered to be a waste and available in huge quantities after frying processes, were directly ethoxylated using a conventional cheap catalyst in order to obtain an economically valuable ethoxylated product to replace the imported intermediate derivatives and at the same time the environment will be rid of one of its pollutants. Therefore, this work was devoted to exploring its application as a fatliquoring agent in the leather industry. Overused sunflower and olein oils were directly ethoxylated using ethylene oxide gas in the presence of 3% KOH catalyst at 180 °C for 20 h. The prepared products were applied as nonionic fatliquors. The fatliquoring process is the operation in which a fatty matter is introduced into the leather fibers. The results obtained showed that the prepared ethoxylated overused oils were effective fatliquors with high HLB values giving stable oil in water emulsion as well as high stability against acid, alkali and different metallic salts. The fatliquored leather had improved mechanical properties such as tensile strength and elongation at break. In addition, a significant enhancement of the texture of the treated leather by the two prepared fatliquors as indicated from the scanning electron microscope images was observed. Also the results indicated that ethoxylated overused sunflower oil gave better results than those of ethoxylated overused olein oil(Nashy, E.-S.H.A.A,2011).

# Chapter Three

# Materials and methods

# Chapter Three

## Materials and methods

## 3.1 Raw materials

Use castor seeds to extract oil, obtained from local market, for use on chrome-tanned leather and re-tanned vegetable tanning materials.

## 3.2 Tools, instruments, machines and apparatuses

- A Soxhlet extractor is a piece of laboratory apparatus, to extract oil.Standard 500ml glass beaker.Great for chemistry experiments.
- Funnel, is a piece of laboratory glassware used in liquid-liquid extractions to separate (partition) the components of a mixture into two immiscible solvent phases of different densities.
- pH meter, to measure acidity in oil.
- Drum, which are fatliquoring process.
- A tensile machine was used for determining the tensile and tears strength of crust leathers.
- Lastometer tester was used for determination of the load and distension at grain crack of crust leathers.

## 3.3 Chemicals

### 3.3.1 Extraction of castor oil

Use the normal hexane, a solvent for the extraction of castor oil seeds. It is after it has been extracted castor oil were added concentrated sulfuric acid (98%) of the sulphated oil. After that, use sodium chloride salt to separate the oil from the acid. The washing with a solution of salt-saturated, and observed the separation of castor oil sulphated component higher layer and the bottom deposits white is sodium sulfate ($Na_2SO_4$) and risinggas$SO_2$byminoritywasto get rid of deposits. Soap has been added to reduce the surface tension in order to increase the solubility of castor oil in the water sulfurated.

### 3.3.2 Preparation of castor seeds for extraction

Castor seeds were cleaned and separated from foreign materials and impurities; sun dried until the casing splits and sheds the seeds. The beans were further dried in the oven at 60°C for 7hrs to a constant weight. The sheds were blown away and separated from the nibs using a tray. Then, the beans were crushed into a paste to rupture the cell walls so as to release castor oil for extraction (Abdelaziz. s.l,2014)

### 3.3.3 Determination of oil content of castor seeds

50 gms of crushed castor bean were placed in a filter paper and inserted in the center of the extractor. 250 ml of normal Hexane were weighed and poured into a round bottom flask. The flask was heated at 68°C. The solvent boiled and vaporized through the vertical tube into the condenser at the top. The condensate dropped onto the thimble in the center containing the solid sample to be extracted. The extract seeped through the thimble into the round bottom flask via the siphon. This was allowed to continue for six hours. At the end of extraction, the sample was then removed from the tube, dried in an oven, cooled in the desiccators and weighed to determine the amount of oil extracted (Abdelaziz. s.l,2014,p)

The % yield of castor oil was calculated as follows:

$$\%yield = \frac{y_1 - y_2}{y_1} \times 100 \quad \text{……..…………………….. (3-1)}$$

Where $y_1$ and $y_2$ are the weights of sample before and after extraction respectively.

### 3.3.4 Determination of Moisture Content of castor seeds

50g of cleaned bean sample was taken and dried in an oven at 80°C, weighed at one hour intervals. The process of drying and weighing was repeated until a constant weight was obtained. The moisture content was calculated as follows: **(Abdelaziz. s.l,2014)**.

$$\%\text{moisture} = \frac{w_1 - w_2}{w_1} \times 100 \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots (3\text{-}2)$$

Where $w_1$ and $w_2$ were the weights of the sample before and after drying respectively.

### 3.3.5 Preparation of fatliquor from castor oil

A concentrated sulphuric acid (98%) was added drop wise to 500 gm of castor oil with a constant stirring at 18-20°C. The sulphation process was carried out slowly for about 3hrs. A saturated sodium chloride solution was added to the resultant products and mixed with them. The mixture was then kept in a separating funnel overnight to separate the layers. The upper layer was neutralized to pH 5.0 by adding 30% sodium hydroxide solution to produce the fatliquor. (Society of leather Technologist and Chemists,1965)

### 3.3.6 Analysis of raw and sulphated castor oil

The raw and sulphated castor oil were subjected to physical and chemical tests. The tests were carried out in Central Laboratory for Technical Services and calibration (CLTSC), by using NIST traceable reference equipment and materials in accordance with ISO/IEC 17025:2005 requirements and the test methods referenced below meets ISO/IEC 17025:2005 and accreditation bodies. The test method used is describing against the testing parameters in the results tables. The reported test results are representing the tested sample only. The tests were carried out at 20°C and 60% relative humidity.

The uncertainties values are calculated according to the standard uncertainty by coverage factor (k = 2) at 95% confidence level according to ISO 17025:2005 and accreditation body requirements as describing in the work instruction CLTS/Work 1./5.4.1(Society of leather Technologist and Chemists,1965)

### 3.3.6.1 Determination of Saponification Value

The indicator method was used as specified in Standard ISO 3657 (1988). 2G of the sample was weighed into a conical flask. Then add 25 ml of 0.1N ethylene hydroxide. The content that was constantly stirred was allowed to boil gently for 60min. A reflux capacitor is placed on the vial containing the mixture. A few drops of phenolphthalene indicator were added to a warm solution and then calibrated with 0.5M HCl to the endpoint until the pink color of the indicator just disappeared. The same procedure was used for other samples and vacuum. V = the size of the solution used in the blank test; the size of the solution used to determine natural N = normal pH of the used hydrochloric acid. M = mass of the sample(Society of leather Technologist and Chemists,1965)

### 3.3.6.2 Determination of Iodine Value

The method specified by ISO 3961 (1989) was used. The weight of 0.4 g of the sample was weighed into a conical flask and 20 mL of tetrachloride carbon tetrachloride was added to dissolve the oil. Then 25ml of the dam detector was added to the flask using a safety pipette in the smoke chamber. Then the stopper was inserted and the content of the vial flask was strongly. Then put the flask in the dark for two hours and 30 minutes. At the end of this period, 20ml of 10% potassium iodide and 125 ml of water were added using a measuring cylinder. The content was calibrated with 0.1M sodium the osulfate solutions until the color disappeared almost yellow. A few drops of 1% starch indicator were added and the calibration continued by adding a wise thiosulfate drop until the blue disappeared after a strong vibration. The same procedure was used to test empty and other samples. V1 = the volume of sodium thiosulfate used in the blank; V2 = the volume of sodium thiosulfate used to determine, M = mass of the sample (V1-V2) / M (Society of leather Technologist and Chemists,1965)

### 3.3.6.3 Determination of Specific Gravity

Density bottle was used to determining the density of the oil. A clean and dry bottle of 25ml capacity was weighed (W0) and then filled with the oil, stopper inserted and reweighed to give (W1). The oil was substituted with water after washing and drying the bottle and weighed to give (W2). The expression for specific gravity (Sp.gr) is: Sp.gr = (W1-W0)/(W2-W0) = Mass of the substance / Mass of an equal volume of water(Society of leather Technologist and Chemists,1965)

### 3.3.6.4 Determination of Viscosity

A clean, dried viscometer with a flow time above 200 seconds for the fluid to be tested was elected. The sample was filtered through a sintered glass (fine mesh screen) to eliminate dust and other solid material in the liquid sample. The viscosity meter was charged with the sample by inverting the tube's thinner arm into the liquid sample and suction force was drawn up to the upper timing mark of the viscometer, after which the instrument was turned to its normal vertical position. The viscometer was placed into a holder and inserted to a constant temperature bath set at 29°C and allowed approximately 10 minutes for the sample to come to the bath temperature at 29°C. The suction force was then applied to the thinner arm to draw the sample slightly above the upper timing mark. The afflux time by timing the flow of the sample as it flow freely from the upper timing mark to the lower timing mark was recorded (Society of leather Technologist and Chemists,1965).

#### 3.3.6.5Determination of Acid Value

25ml of diethyl ether and 25ml of ethanol was mixed in a 250ml beaker. The resulting mixture was added to 10g of oil in a 250ml conical flask and few drops of phenolphthalein were added to the mixture. The mixture was titrated with 0.1M NaOH to the end point with consistent shaking for which a dark pink colour was observed and the volume of 0.1M NaOH (V0) was noted. Free Fatty Acid (FFA) was calculated as: V0/W0·2.82·100, where 100ml of 0.lM NaOH = 2.83g of Oleic acid, $W_0$ = sample weight (Society of leather Technologist and Chemists,1965)

### 3.3.7 Retanning and fatliquoring process

Full grain chrome tanned bovine leathers of fairly similar size and free from physical defects leathers were wet back, retanned, fatliquored and converted into crust leather using the recipe presented in Table 2.1. The prepared sulphated castor oil was used for experimental leathers and an imported fatliquor was used for control leathers. The process was carried out using an experimental stainless steel drum (Society of leather Technologist and Chemists,1965)

**Table 3.1** Formulation of the retanning processes for experimental and control leathers

| Process/products | % | Time (min) | pH | Remarks |
|---|---|---|---|---|
| Wetting back | | | | |
| water | 200 | 30 | | |
| Ps/A (wetting agent) | 0.3 | | | |
| Formic acid | 0.3 | | | |
| Drain | | | | |
| Re-chroming | | | | |
| Water | 200 | | | |

|  |  |  |  |  |
|---|---|---|---|---|
| chrome | 2 |  |  |  |
| Leave O/N, run 10′ morning, drain, wash |  |  |  |  |
| Neutralization |  |  |  |  |
| Water | 150 |  |  |  |
| KNB( Syntan) | 1 | 30 |  |  |
| Sod-formate | 2 |  |  |  |
| Sod- bicarbonate | 1 | 60 | 5 |  |
| Drain,  wash |  |  |  |  |
| Retanning |  |  |  |  |
| Water | 200 |  |  |  |
| Mimosa | 3 |  |  |  |
| (syntan) | 3 | 40 |  |  |
| (syntan) | 3 |  |  |  |
| Coraline 6 | 1.5 | 10 |  |  |
| syntan | 1.5 | 30 |  |  |
| syntan | 1.5 |  |  |  |
| Fatliquoring |  |  |  |  |
| Filler | 3 |  |  |  |
| Fatliqour | 6 | 45 |  |  |
| Formic acid | 1.5 | 40 |  |  |
| Drain, wash, pile O/N, toggle dry, trim and stake<br>*Percentages are based on wet blue weight. |  |  |  |  |

## 3.4 Reactor design to produce sulphated castor oil

The reactor design contains a mixer and a jacket through which passes cold water works heat exchanger to cool the reactor, diameter of jacket tenth of the diameter reactor is controlled in the water flow through the wrap, which is on the way to control the temperature of water in order to control heat required.

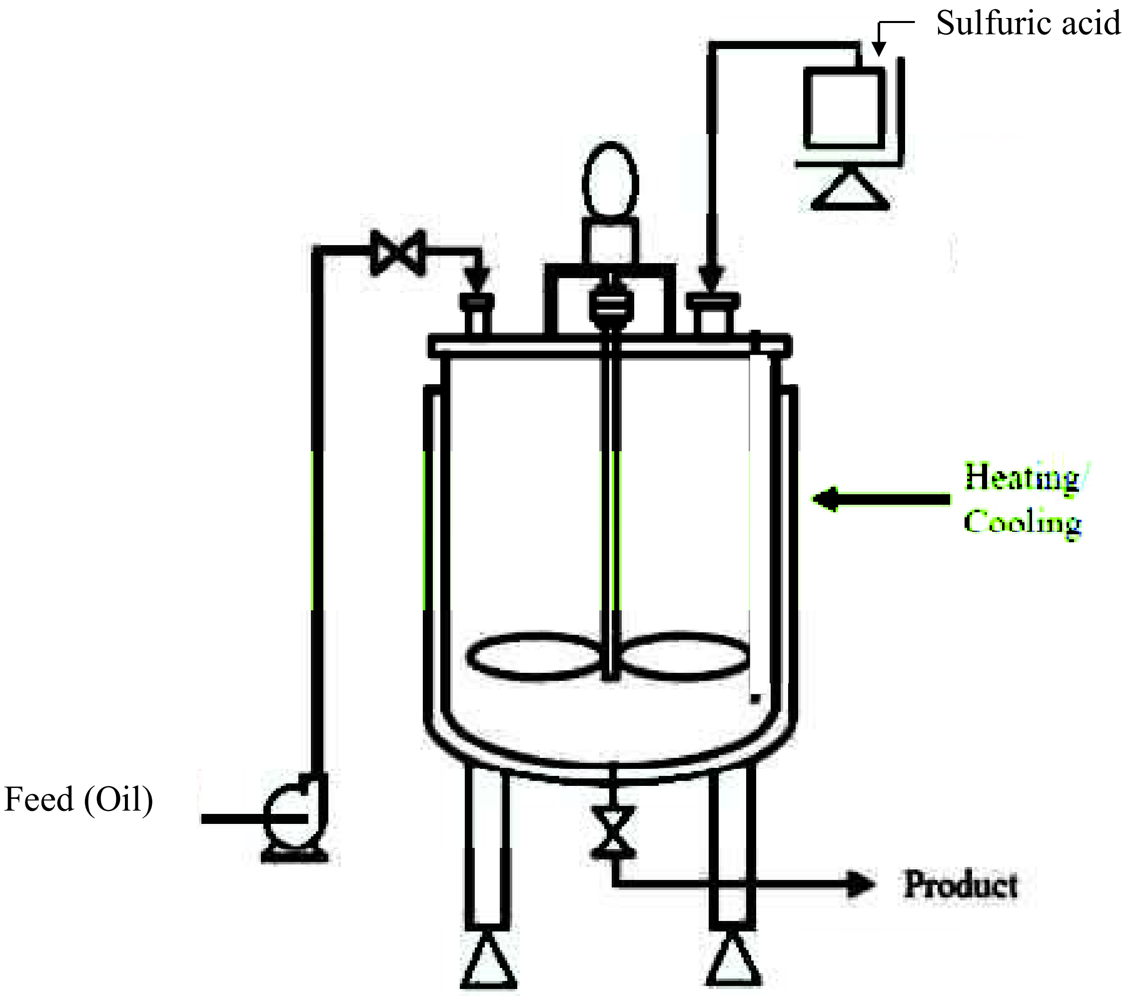


**Figure 3.1** Reactor of sulphated castor oil

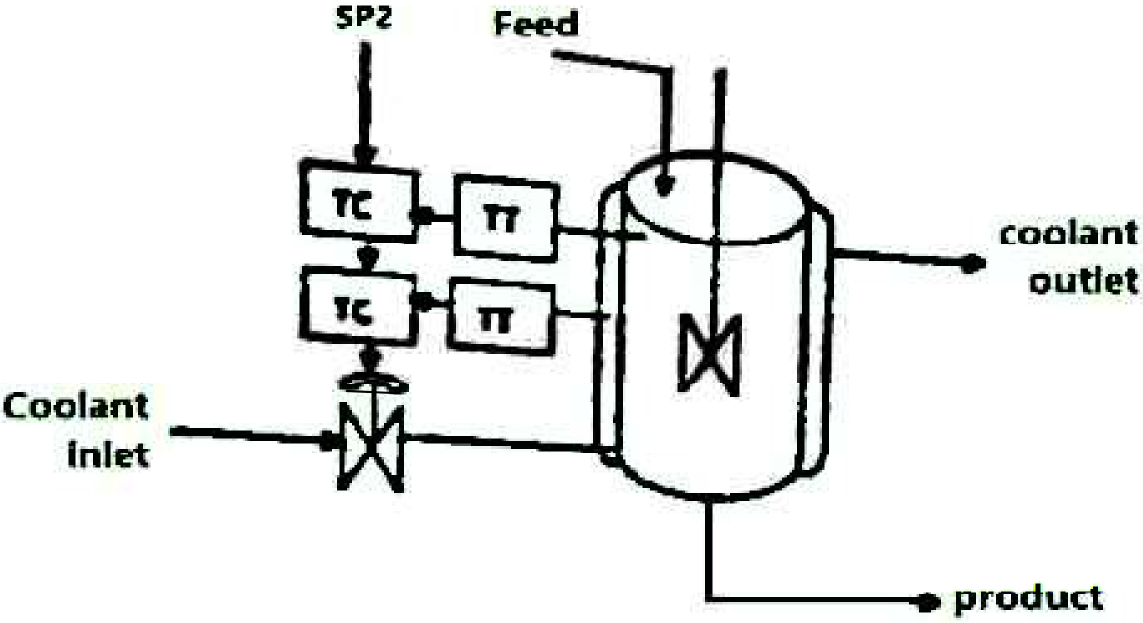


**Figure 3.2** control circuit

## 3.5 Physical testing of leathers

Tensile carried out load in crack grain leather crust, according to official methods of analysis (Society of leather Technologist and Chemists,1965)

### 3.5.1 Sample preparation:

The specimens for physical testing were obtained from experimental and control crust leathers, cut from official position (**Figure 3.4**)

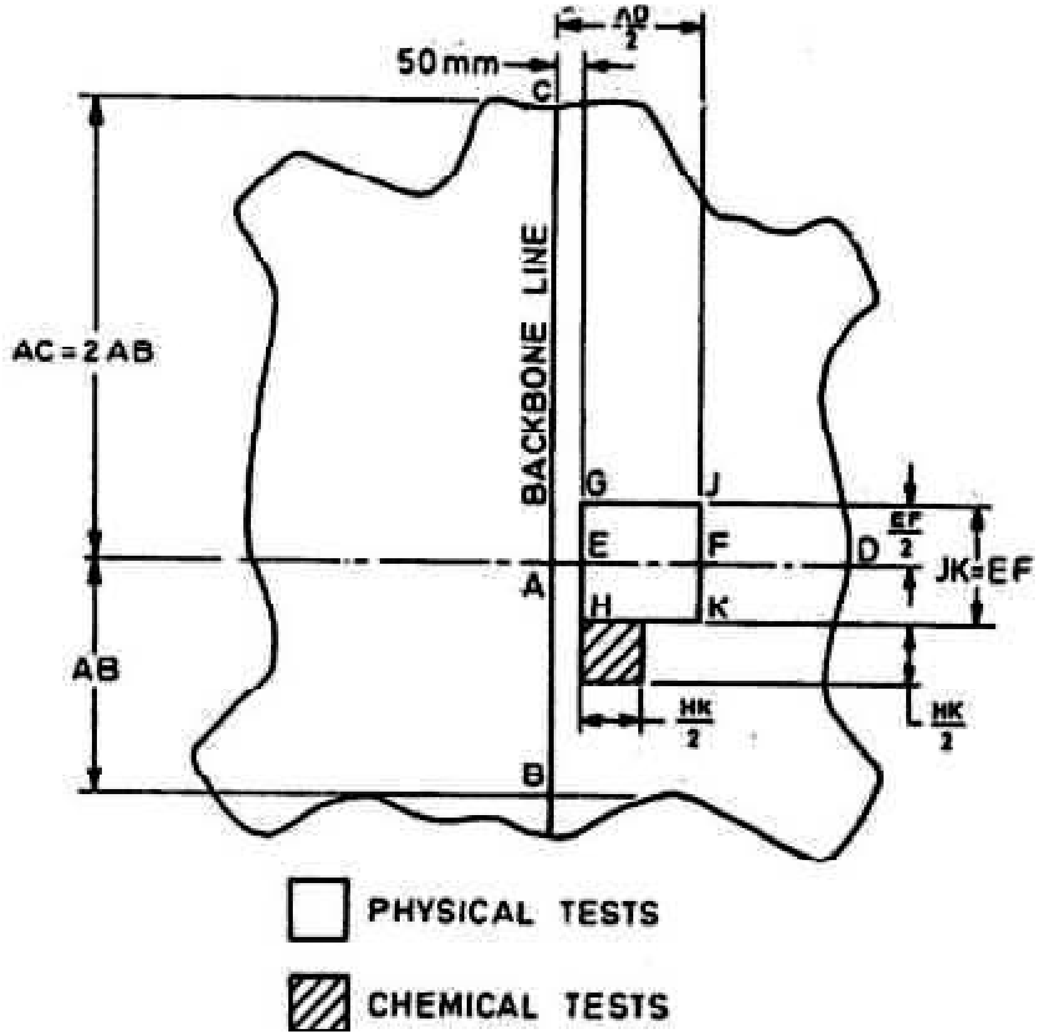


**Figure3.3**: Sample locations of physical and chemical test

location for cutting out test pieces in a whole hide, skin and side Specimens for tensile and tear strength were cut parallel and perpendicular to the backbone and the thickness of each specimen was measured using a standard thickness gauge at three positions on the grain side and three positions on the flesh side, and then the mean of the six measurements was calculated. Each value for tensile and tear strength represents the average of four samples (2values along the backbone and 2 values across the backbone). Each value for grain crack strength is the average of three samples (Society of leather Technologist and Chemists., Official Methods of Analysis,1965).

Represents a skin or hide with the head removed. B is the root of the tail. A is a point on the backbone such that AC = 2AB. AD is a line perpendicular to BC. F is the midpoint of AD, and AE is of length 50mm.the lines GH and JK, whose midpoints are E and F respectively, are parallel to BC and each of length equal to EF. The pieces for physical tests were cut from the square HKJG, and as close to the line EF as possible (Society of leather Technologist and Chemists,1965)

### 3.5.2 Conditioning:

During the 48 hours immediately preceding its use in a test, each specimen for physical testing were kept in a standard atmosphere of temperature 20º ± 2ºC and relative humidity 65 ± 2 %( Society of leather Technologist and Chemists,1965)

### 3.5.3 Measurement of thickness:

After the specimens were conditioned in accordance with the method for conditioning, the specimens were placed in the gauge with the grain side up.

The load is applied gently and the reading is taken five seconds after the full load is reached. Three measurements were taken at three positions; at the point E and midway between E and the lines AB, CD and the mean of the three thickness measurements was taken as the thickness of the specimen. The area of cross section of each specimen was calculated by multiplying its width by its thickness (Society of leather Technologist and Chemists,1965)

### 3.5.4 Measurement of tensile strength and percentage:

Four specimens were cut, (two parallel and two perpendicular to the backbone) with the press knife having the shape and dimensions of the internal surfaces shown in **figure 3.5**

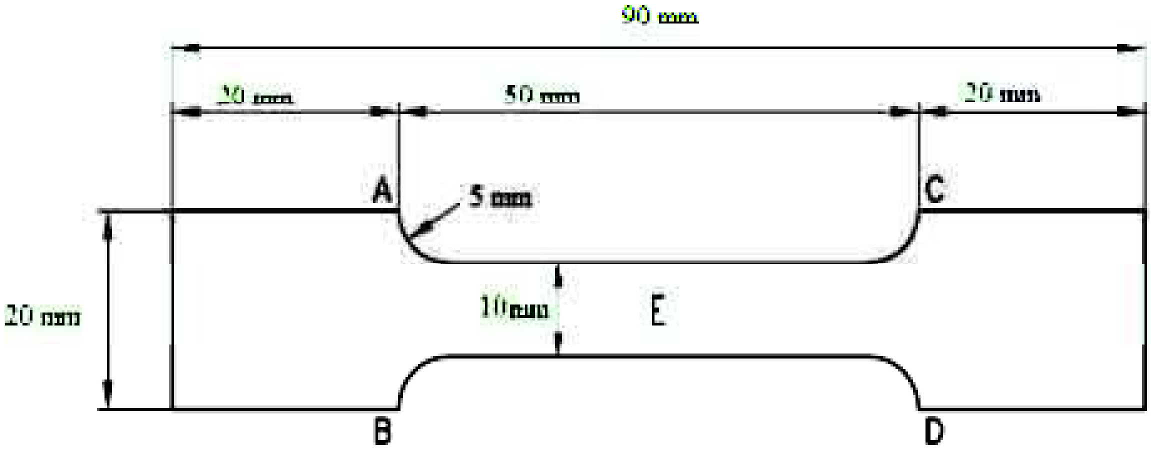


**Figure 3.4:** Shape and dimensions of tensile strength specimen

The specimens were conditioned according to the method of conditioning.
The thickness of each specimen is measured to the nearest tenth of a millimeter at three positions on the grain and at the three positions on the flesh side; in each group of three, three measurements were made one at the midpoint E of the waist of the specimen and the other two at positions approximately midway between E and the lines AB, CD. The arithmetic mean of the six measurements is taken as the width of the specimen. The thickness of each specimen is measured in accordance with the method of measurement of thickness. The area of cross section of each specimen was calculated by multiplying its width by its thickness. The jaws of the tensile strength machine were set 100mm apart and the specimen was clamped in the jaws so that the edges of the jaws lie along the lines AB, CD and the grain surface of the specimen lead in one plain. The machine was run until the specimen broken and the highest load reached was taken as the breaking load. The tensile strength was calculated in kg/cm2 by dividing the breaking load by the area of cross- section of the specimen (Society of leather Technologist and Chemists,1965)

### 3.5.5 Measurement of tearing load

Tongue tearing is a simpler tear test where the tearing takes place through the weakest region of the leather specimen. A 5mm diameter hole was punched at a point one- third of the length of the specimen, 75×25mm from one end and on the central line of the specimen as shown in **figure 3.6**

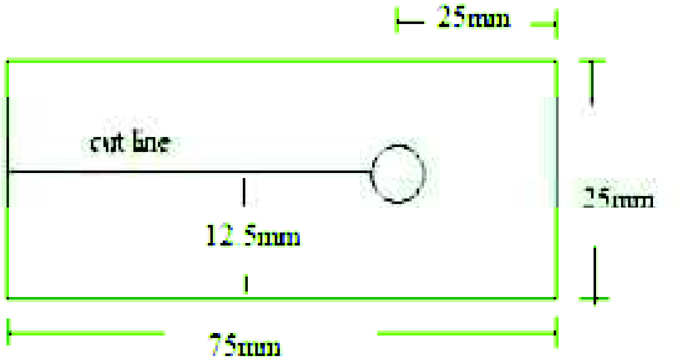


**Figure 3.5:** Tear specimen dimensions

The leather specimen was then cut into two tongues at right angle to the grain surface for two third of its length. The ends of the two tongues thus formed were clamped to the two jaws respectively of the tensile strength tester. The load required to continue the tear is recorded from the dial of the machine (Dutta, S.S,1990,p)

$$\text{Tearing strength kg/cm} = \frac{\text{Tearing load (Kg)}}{\text{Leather thickness}} \ldots\ldots\ldots\ldots\ldots \quad (3\text{-}3)$$

### 3.5.6 Measurement of distension and load at grain crack:

The specimens were cut and conditioned in accordance with method SLP.3.

The conditioned specimen was clamped in the instrument with its flesh surface adjacent to the ball, and its grain surface flat. The distension was increased until the grain cracked then the load and distension was noted (Society of leather Technologist and Chemists,1965)

The loading continued with a little delay as possible, when the disc burst the load and distension was noted. The means of three tests were taken for the load and distension at grain crack and the corresponding values at burst were reported (Society of leather Technologist and Chemists,1965)

## 3.6 Chemical analysis of leathers:

moisture content, chromic oxide content, oil and fats content, total ash content, water soluble matter, insoluble ash, hide substance and degree of tannage of both combined tanned leathers and control were determined by the official methods of analysis(Society of leather Technologist and Chemists,1966)

### 3.6.1 Sampling:

The items envisaged for chemical examination are first divided in accordance with the sampling direction for physical tests see (**figure 3.5**).

The designations given in the figures in SLP.2 also apply on to the present directions. Slight defects in the grain or flesh side are of no significance.

A square piece of leather adjacent to line HK see **(figure 3.5)** in the direction of the root of the tail, is taken from point H and with the side length equal to the half-line HK. If necessary, the section can also be taken from the corresponding place in the opposite half of the skin (Society of leather Technologist and Chemists,1966)

### 3.6.2 Determination of moisture content in leather:

Sample was cut into small pieces and 5gms of the sample were taken and dried at 102±2°C in a normal oven for 5 hours. After cooling in the desiccators, the sample was weighed. After a further one hour drying and half hour cooling, the weight was checked again and the process of drying and cooling was repeated until a constant weight was obtained (Society of leather Technologist and Chemists,1966)

$$\text{moisture}\ \% = \frac{W_1 - W_2}{W_1} \times 100 \ldots\ldots\ldots\ldots (3\text{-}4)$$

$W_1$=weight of sample before drying

$W_2$=weight of sample after drying

### 3.6.3 Determination of oils and fats:

5gm of the sample was placed in a Soxhlet apparatus attached to a weighed flask to be extracted with light petroleum ether B.P bellow 60°C for 5 hours. After extraction, the solvent was decanted from the fatty residue. Then the extract was dried at 100 °C in an oven for 4 hours, cooled and weighed (Society of leather Technologist and Chemists,1966)

$$\text{fat content } \% = \frac{\text{gram extract}}{\text{gram original sample}} \times 100 \text{ .......... (3-5)}$$

### 3.6.4 Determination of total ash:

g of the prepared sample was put into a previously weighed tared crucible, heated gently at first until the leather completely decomposed then, the ashing was continued in a muffle furnace at 800°C until all the carbon is consumed. Then the crucible was cooled in a desiccator and weighed. Reignited, cooled and weighed until constant weight was obtained (Society of leather Technologist and Chemists,1966)

$$\text{total ash in } \% = \frac{\text{gram ash}}{\text{gram original sample}} \times 100\text{........... (3-6)}$$

# Chapter Four

# Results and discussion

# Chapter Four

## Results and discussion

## 4.1 Results

### 4.1.1 Content of castor oil in castor seeds

The average percentage of castor oil extracted from castor seeds is shown in Table4.1.

**Table4.1:** Average content of castor oil in castor seeds

| Sample No. | Weight of sample (gm) | Weight of oil extracted (gm) | % oil content | % average content |
|---|---|---|---|---|
| 1 | 313 | 88.9 | 28.4 | 28.6 |
| 2 | 313 | 89.6 | 28.6 | |
| 3 | 313 | 89.9 | 28.7 | |

### 4.1.2 Physicochemical parameters of raw castor oil

The physicochemical properties of raw and sulphated castor oil are presented in Table4.2 and Table4.3 respectively.

**Table4.2**: Physicochemical parameters of raw castor oil.

| No. | Parameter | Test method No. | Result | Unit |
|---|---|---|---|---|
| 1 | Acid value | AOAC | 1.466 | mgKOH/100gm |
| 2 | Free fatty acid | AOAC | 0.733 | % |
| 3 | Specific Gravity | AOAC2000 | 0.956 | g/cm$^3$ |
| 4 | Viscosity@40°C | Cock and van(1966) | 0.9 | N s/m$^2$, Pa s |

| | | | | |
|---|---|---|---|---|
| 5 | Saponification | BS | 184.59 | Mg/g |
| 6 | Iodine Value | AOAC | 85.6 | [g $I_2$ /100g of Oil] |
| 7 | Solubility in water | - | soluble | - |

### 4.1.3 Physicochemical properties of sulphated castor oil

**Table4.3:** Physicochemical properties of sulphated castor oil

| No. | Test | Test method No. | Result | Unit |
|---|---|---|---|---|
| 1 | Acid value | AOAC | 0.82 | mgKOH/100gm |
| 2 | Free fatty acid | AOAC | 0.414 | % |
| 3 | Specific Gravity | AOAC2000 | 0.95714 | g/cm$^3$ |
| 4 | Viscosity@40°C | Cock and van(1966) | 2.43 | N s/m$^2$, Pa s |
| 5 | Saponification | BS | 181.2 | Mg/g |
| 6 | Iodine Value | AOAC | 82.4 | [g $I_2$ /100g of Oil] |
| 7 | Solubility in water | - | soluble | - |

### 4.1.4 ASTM properties of quality castor oil

**Table4.4:** ASTM properties of quality castor oil (2)

| **Property** | **Range** | **Selected** |
|---|---|---|
| Specific gravity 20/25 °C | 0.957 – 0.968 | 0.962 |
| Saponification value | 175 – 187 | 181 |
| Iodine value | 82 – 88 | 85 |
| Acid value | 0.4 – 4.0 | 3 |
| Viscosity at 40 °C cSt | 240.12 | - |
| Viscosity at 100 °C cSt | 20.00 | 20.00 |

| | | |
|---|---|---|
| Viscosity index | 90.00 | 90.00 |
| Flash point | 320 °C | 320 °C |
| Pour point | -21.7 °C | -21.7 °C |
| Free fatty acid | 0.3–0.7 | 0.5 |

### 4.1.5 Physical Testing Results for Experimental and Control Upper Leathers

**Table 4.5:** Physical Testing of Experimental and Control Upper Leathers

| Test | Average Readings | |
|---|---|---|
| | Experimental | Control |
| Thickness (mm) | 2.1 | 2.0 |
| Tensile strength (kg/cm2) | 334.8 | 330.5 |
| Elongation at break % | 59 | 52 |
| Single hole tear strength (kg/cm) | 137 | 138 |
| Tongue tear strength (kg/cm) | 40.5 | 49 |
| Double hole tear strength (kg/cm) | 178.5 | 280 |
| Distention at grain crack (mm) | 13.3 | 12.0 |
| Flexibility | Pass | pass |

### 4.1.6 Chemical Analysis of Experimental and Control Upper Leathers

**Table 4.6** Chemical Analysis of Experimental and Control Upper Leathers

| Test | Readings Average | |
|---|---|---|
| | Experimental | Control |
| Moisture content % | 5.97 | 6.9 |
| Fat and Oil % | 8.50 | 10.9 |
| Ash content % | 7.11 | 8.7 |

## 4.2 Discussion

Using hexane as a solvent, the average value of oil content in castor beans was found to be 28.6% it is in the near range between30 -55% obtained in the literature by(Abdelaziz. s.l,2014,p2) using the same solvent. According to Nangbes, J. extraction with hexane gave the lowest yield when it was compared with isopropanol. The high yield may be due to environmental factor which enhance the growth and productivity of the seed (Nangbes, J. G,2013,p105). The mode of extraction is a very important parameter affecting the yield. It is reported that the best available method for extraction of castor oil is by hydraulic pressing (Nangbes, J. G,2013).

Table 4.2 presents the physical properties obtained for the crude castor oil. The specific gravity value was found to be 0.956 g/cm$^3$it is agree with the ASTM standards properties of quality castor oil reported by Nangbes, J. G that is 0.962.

The viscosity of the crude castor oil was determined at 40°C and it was found to be 0.9N s/m$^2$, Pa s. As can be seen the value of viscosity obtained for crude oil was significantly higher than that of ASTM standard reported in the literature Nangbes, J. G.This may be attributed to the presence of some impurities and components.

The chemical properties analysis shown in table 4.2 and table 4.3 indicates that the acid value of crude oil and sulphated oil is 1.466mgKOH/100gm and 0.82mgKOH/100gm respectively of oil. This result is slightly less than the values of 3mg KOH/g of Oil obtained by (U. G. AKPAN,etal,2006) however, it falls within the standard range of 0.40-4.0 specified in literature.

Table 4.3 shows the results for the saponification value of the crude and refined oil that were found to be 181.2mgKOH/g of oil, it is agree with the ASTM standards properties of quality castor oil reported by U. G. AKPAN that is 181. However, all of them are highly comparable with ASTM specification for quality castor oil. The free fatty acid was determined to be 0.414; this result is considered within the standard and in the permissible range (U. G. AKPAN,etal,2006).From4.5 the table it was observed that the strength properties of experimental leather were comparable to the strength properties of the control leathers. The results obtained were found within the limits of standard specification. The results show that the prepared fatliqoure has good lubrication properties and possesses good penetration power and emulsion stability.

The high value of tensile strength indicates the high strength of collagen fibers.The elongation at break indicating the softness, flexibility, strength and toughness of eather. The experimental leathers pass the flexibility test while the control leather pass.This shows the good lubrication and penetration properties of the prepared fatliquor.

From table 4.6the chemical characteristics of the experimental and control leathers were found to be quite normal. The moisture, fat and ash content, in leather seem to have acceptable value for both the experimental and control leathers.

# Chapter Five

# Conclusion and recommendations

# Chapter Five

## Conclusion and recommendations

### 5-1 Conclusions

It is concluded that sulphated castor oil is suitable as a fatliquoring material of all types of leathers. Castor oil is available, cheap, toxic to human and nonedible.

The physical and chemical characteristics of leathers produced and treated with the prepared fatliquor are comparable to that of leathers produced using the imported fatliquor.and locally produced fatliquor from castor oil would be a substitute to imported fatliquors.

### 5.2 Recommendations

1- It is recommended to make leathers that can be fatliquored with sulphated castor oil that the physical and chemical characteristics of the leathers processed by the prepared castor oil fatliquor are comparable to that of leathers produced using imported fatliquor.

2- A plantation of castor oil plant needs to be made for castor oil wood industry.

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# USING CHARACTERIZATION AND SYNTHESIS OF FATLIQUOR FROM SUDANESE CASTOR OIL

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## ABSTRACT

Castor oil is classed as semi-drying oil because it oxidizes on exposure to air, giving a gummy film, and can be highly sulphated to make it miscible in water. In the raw condition it is seldom used in fatliquoring of leather because the softness and flexibility of the leather will decrease on storage as the castor oil dries. Castor seeds were collected, sundried, and crushed. Castor oil was extracted in a soxhlet apparatus using hexane as a solvent. . A yield of 90%was obtained. Physical and chemical analyses were carried out for the obtained crude oil. The crude oil was converted to fatliquor by sulphation process using sulphuric acid. The physical and chemical analyses were carried out for the produced fatliquor and compared with the standards.This paper carried out experimental study, through extraction and characterization of castor oil. Normal hexane was used as a solvent for the extraction process. The characterization analysis revealed that tested parameters, which include specific gravity, refractive index, acid, saponification and iodine values for both crude and refined castor oil produced, were within the ASTM standard specifications.

**KEYWORDS**: Castor oil, fatliquoring . extraction , characterization analysis

## INTRODUCTION

Castor plant (Ricinuscommunis), is grown in tropical and warm temperate regions throughout the world. It grows naturally over a wide range of geographical regions and may be activating under a variety of physical and climatic regions. (1)

Castor oil is a vegetable oil obtained from the castor bean (or castor seed) that has a characteristic structure and many uses. It is obtained by pressing and solvent extraction. Castor oil is colorless to very pale yellow liquid with mild or no odor or taste. Its boiling point is 313°C (595° F) and its density is 961 kg/m3. It is a triglyceride in which approximately 90 percent of fatty acid chains are ricinoleate. Oleate and linoleates are the other significant components. (2)

Like any other vegetable oils and animal fats, it is a triglyceride, which chemically is a glycerol molecule with each of its three hydroxyl group esterified with a long clown fatty acid. Its major fatty acid is the unsaturated, hydroxylated 12-hydroxy, 9-octadecenoic acid, known familiarly as Ricinoleic acid. The fatty acid composition of a typical castor oil contains about 87% of ricinoleic acid. (3; 1) It is reported that castor beans contains about 30-35% oil. (3). And has one of the highest viscosities among vegetable oils, with a molecular weight of 298. (1)

Castor oil can be extracted from castor beans by either mechanical pressing or solvent extraction or combination of the two. (4) (3) (2).

In mechanical pressing, the seeds are crushed and then adjusted to low moisture content by warming in a steam-jacketed vessel. Then, the crushed seeds are loaded into a hydraulic press and pressed by mechanical means to extract oil. The resulting oil has a light colour and low free fatty acids. Mechanical pressing only recovers about 45% of oil from the beans and the remainder in the cake can be recovered by solvent extraction. The crushed seeds are extracted with a solvent in a Soxhlet extractor or commercial extractor. Solvents used for extraction include heptane, hexane and petroleum ethers. (4)

However, castor oil and its derivatives are used in the production of paints, varnishes, lacquers, and other protective coatings, lubricants and grease, hydraulic fluids, soaps, printing inks, linoleum, oil cloth and as a raw material in the manufacturing of various chemicals sebacic acid and undecylenic acid, used in the production of plasticizer and Nylon (3). Also castor oil has a special use as a "softener" in nitrocellulose finishes. As opposed to plasticizer oils it gives softness without stickiness or tackiness (5).

The presence of a hydroxyl group at C12 of the recinoleic acid, the ester linkages, double bonds and hydroxyl groups provide reaction sites that makes it usually polar, which provide sites for the production of a wide range of natural and synthetic resins, waxes, polymers and elastomers. It also has excellent emollient and lubricating properties, a marked ability to wet and disperse dyes, pigments and fillers as well as several medicinal values. Castor oil has excellent solubility in methanol and hence theoretically an ideal oil for transesterification to biodiesel, requiring a minimum amount of catalyst and heating which can reduce cost for production. and also allows chemical derivation that is not practical with most other seed oils.

In the dehydration process or sulphonation carried out at about 250 °C in the presence of catalysts such as concentrated sulphuric acid or activated earth, and under an inert atmospheric condition or vacuum, The hydroxyl group and an adjacent hydrogen atom from the C-11 or C-13 position of the ricinoleic acid portion of the molecule are removed as water. This yield a mixture of two acids each containing two double bonds that results in oil called Turkey-red oil having the properties of tung oil. Thus, the oil can be used in the production of vanishes, lacquers, protective coatings, lubricants, soaps, cosmetics, paints, inks, and it is a primary raw material for the production of nylon and other synthetic resins and fibers and a basic ingredient in racing motor oil for high-performance automobile motorcycle engines.

The castor meal or cake is mainly used as fertilizer; this is because it is unsuitable as an animal feed because of the presence of toxic protein called ricin and toxic allergen often referred to as castor bean allergen (CBA). However, it is noteworthy that none of the toxic components is carried into the oil. (1)

Castor oil has been used on the manufacturing of more than 800 products, ranging from bullet-proof glasses, contact lenses, lipsticks, metal soaps, special engine and high rotation reactors lubricants, high resistance plastics, and polyurethanes. . (Musa, U 2015)

Castor oil is used either in its crude form, or in the refined hydrogenated form. Typically, 65% of it is processed, 28% is refined, 12% is hydrogenated, 20% is dehydrated, and 5% is processed to manufacture other derivatives. Castor oil is used as a raw material for paints, coatings, inks, lubricants and a wide variety of other products. (4)

Castor oil has one double bond in each fatty acid chain and is classified as non-drying oil. Like all other vegetable oils, it has different physical and chemical properties that vary with the method of extraction. Relative to other vegetable oils, it has a good shelf life and does not turn rancid unless subjected to excessive heat. Cold-pressed castor oil has low acid and iodine values, a slightly higher saponification value compared to that extracted using solvent extraction as well as being lighter in colour. In literature, various attempts were made to optimize castor oil recovery. The optimum results were a recovery and moisture content of 48.75% and 5.8% respectively. The objective of this study was to optimize castor oil recovery from castor beans obtained from Mpumalanga Province of South Africa. (4)

This oil is unique among vegetable oils and its uniqueness is derived from the presence of a hydroxyl fatty acid known as ricinoleic acid (12-hydroxyl-cis-9-octadecenoic acid) which constitutes about 90% of the total fatty acids of the oil. Castor oil is also distinguished from other vegetable oils by its high specific gravity, thickness and hydroxyl value. (4)

This work is however aimed at extraction and characterization of the castor seed oil, through the Extraction of castor oil from castor bean by solvent extraction process; determination of physicochemical parameters and fatty acid composition of the castor seed oil extract. Results obtained shall be X-rayed critically with the aim of bringing out the industrial potentials of the oil for economic gains. (1)

As we know, physical-mechanical properties and sensory properties are two important parameters for evaluating the application performance of leather and leather products. Softness is especially one of the most important physical properties to be taken into consideration when assessing the quality of light leathers. Among leather chemical additives, fatliquoring agents are one of the most effective chemicals to impart softness and flexibility to leather and their use is critical to attaining the required characteristics for leather and leather products. Fatliquoring is an oil-addition process by which the leather fibres are lubricated so that after drying they will be capable of slipping over one another and produce an adequate compliance and softness. Among leather chemical additives, fatliquoring agents are one of the most effective chemicals to impart softness and flexibility to leather and their use is critical to attaining the required characteristics for leather and leather products. Fatliquoring is an oil-addition process by which the leather fibres are lubricated so that after drying they will be capable of slipping over one another and produce an adequate compliance and softness.(3)

In the process, fatliquors penetrate into the gaps of collagen fibres; their polar groups attract each other, and form a lubricant film around the collagen fibres.( 5)Fatliquors in leather collagen networks can be regarded as plasticizers of leather materials. (6)

Leather making processes usually require the addition of fatliquoring agents to separate the leather fibers from each other for promoting leather hand feeling and softness. The fatliquoring agents are mainly chemically modified products of animal and vegetable oils.

In order to produce soft leather, after tanning the leather is processed through a fatliquoring step, which is designed to introduce oils and fats into the leather matrix preventing the adhesion of fibers. Fatliquoring is one of the critical steps for garment and upholstery leather manufacturing, which makes the leather soft and has a pleasant feel. The physical characteristics of the leather, as well as comfort properties of the leather, depend on fatliquoring. The term fatliquoring agents, which are used for bringing oils and fats into the leather-fiber network, are emulsifying mixtures. Emulsification of fatliquoring agent is usually achieved by introduction of phosphate, sulfonate and sulfite groups, etc. into the structure of oils and fats or by addition of surfactants to the composition of fatliquoring agent. Although any oil or fat can be used as raw material for producing fatliquoring agent, the most commonly used oils and fats are those of neat foot oil, cod oil, sperm oil, castor oil, coconut oil, rapeseed oil and palm oil, etc. These have a similar basic chemical structure consisting of triglycerides. (7)

Lubricants or fatliquors are applied to the leather to keep the fibres apart during drying and to reduce frictional forces within the fibre weave. Proper lubrication or fatliquoring is necessary to obtain leather with requisite characteristics. This process protects the leather against cracking since it prevents the adhesion of the fibres during drying. The main characteristics of fatliquored leathers are feel, softness and a certain degree of water repellency. Physical properties such as tear resistance, break, and tensile strength as well as comfort properties of leathers depend on fatliquoring. The fatliquoring process introduces oils and fats into the leather matrix in finely dispersed form. This is attained by emulsification process through introduction of sulphate and sulphonate groups into the structure of oils and fats or through addition of surfactants to the composition of fatliquors. Fatliquor emulsions are also prepared by exposed natural and sulphated fats to ultrasonic weaves. (8)

Fatliquor affects the physical properties of the leather and makes more flexible and softer. (9)

## MATERIALS AND METHODS

### Experimental study

Castor seeds were cleaned and separated from foreign materials and impurities, sun dried until the casing splits and sheds the seeds. The beans were further dried in the oven at 60°C for 7hrs to a constant weight. The sheds were blown away and separated from the nibs using a tray. Then, the beans were crushed into a paste to rupture the cell walls so as to release castor oil for extraction (2)

Determination of oil content of castor seeds

50 gms of crushed castor bean were placed in a filter paper and inserted in the centre of the extractor. 250 ml of normal Hexane were weighed and poured into a round bottom flask. The flask was heated at 68°C. The solvent boiled and vaporized through the vertical tube into the condenser at the top. The condensate dropped onto the thimble in the centre containing the solid sample to be extracted. The extract seeped through the thimble into the round bottom flask via the siphon. This was allowed to continue for six hours. At the end of extraction, the sample was then removed from the tube, dried in an oven, cooled in the desiccators and weighed to determine the amount of oil extracted (4).

The % yield of castor oil was calculated as follows:

$$\%\text{yield} = \frac{y_1 - y_2}{y_1} \times 100$$

Where $y_1$ and $y_2$ are the weights of sample before and after extraction respectively.

Determination of Moisture Content of castor seeds

50g of cleaned bean sample was taken and dried in an oven at 80°C, weighed at one hour intervals. The process of drying and weighing was repeated until a constant weight was obtained. The moisture content was calculated as follows: (4)

$$\%\text{moisture} = \frac{w_1 - w_2}{w_1} \times 100$$

Where $w_1$ and $w_2$ were the weights of the sample before and after drying respectively.

Preparation of fatliquor from castor oil

A concentrated sulphuric acid (98%) was added drop wise to 500 gm of castor oil with a constant stirring at 18-20°C. The sulphation process was carried out slowly for about 3hrs. A saturated sodium chloride solution was added to the resultant products and mixed with them. The mixture was then kept in a separating funnel overnight to separate the layers. The upper layer was neutralized to pH 5.0 by adding 30% sodium hydroxide solution to produce the fatliquor.

Analysis of raw and sulphated castor oil

The raw and sulphated castor oil were subjected to physical and chemical tests. The tests were carried out in Central Laboratory for Technical Services and calibration (CLTSC), by using NIST traceable reference equipment and materials in accordance with ISO/IEC 17025:2005 requirements and the test methods referenced below meets ISO/IEC 17025:2005 and accreditation bodies. The test method used is describing against the testing parameters in the results tables. The reported test results are representing the tested sample only. The tests were carried out at 20°C and 60% relative humidity.

The uncertainties values are calculated according to the standard uncertainty by coverage factor (k = 2) at 95% confidence level according to ISO 17025:2005 and accreditation body requirements as describing in the work instruction CLTS/Work 1./5.4.1

## RESULTS AND DISCUSSION

The average percentage of castor oil extracted from castor seeds is shown in **Table1**.

***Table1:Average content of castor oil in castor seeds***

| SAMPLE NO. | WEIGHT OF SAMPLE (GM) | WEIGHT OF OIL EXTRACTED (GM) | % OIL CONTENT | % AVERAGE CONTENT |
|---|---|---|---|---|
| **1** | **313** | **88.9** | **28.4** | **28.6** |
| **2** | **313** | **89.6** | **28.6** | |
| **3** | **313** | **89.9** | **28.7** | |

The physicochemical properties of raw and sulphated castor oil are presented in **Table2** and **Table3** respectively.

***Table2: Physicochemical parameters of raw castor oil.***

| No. | Parameter | Test method No. | Result | Unit |
|---|---|---|---|---|
| 1 | **Acid value** | **AOAC** | **1.466** | mgKOH/100gm |
| 2 | **Free fatty acid** | **AOAC** | **0.733** | % |
| 3 | **Specific Gravity** | **AOAC2000** | **0.956** | g/cm$^3$ |
| 4 | **Viscosity@40˚C** | **Cock and van(1966)** | **894.67** | Cp |
| 5 | Saponification | BS | 184.59 | Mg/g |

***Table3: Physicochemical properties of sulphated castor oil***

| No. | Test | Test method No. | Result | Unit |
|---|---|---|---|---|
| 1 | **Acid value** | **AOAC** | **0.82** | mgKOH/100gm |
| 2 | **Free fatty acid** | **AOAC** | **0.414** | % |
| 3 | **Specific Gravity** | **AOAC2000** | **0.95714** | g/cm$^3$ |
| 4 | **Viscosity@40˚C** | **Cock and van(1966)** | **2.43** | cps |
| 5 | Saponification | BS | 143.5 | Mg/g |

***Table4: ASTM properties of castor oil** (2)*

| Property | Range | Selected |
|---|---|---|
| Specific gravity 20/25 °C | **0.957 – 0.968** | 0.962 |
| Saponification value | **175 – 187** | 181 |
| Iodine value | **82 – 88** | 85 |
| Acid value | **0.4 – 4.0** | 3 |
| Viscosity at 40 °C cSt | **240.12** | - |
| Viscosity at 100 °C cSt | **20.00** | 20.00 |
| Viscosity index | **90.00** | 90.00 |
| Flash point | **320 °C** | 320 °C |
| Pour point | -21.7 °C | -21.7 °C |

Using hexane as a solvent, the average value of oil content in castor beans was found to be 28.6%, less than the range value of 30 -55% obtained in the literature by (2) (3) (1) using the same solvent. According to (1), extraction with hexane gave the lowest yield when it was compared with isopropanol. The high yield may be due to environmental factor which enhance the growth and productivity of the seed (1). The mode of extraction is a very important parameter affecting the yield. It is reported that the best available method for extraction of castor oil is by hydraulic pressing (1).

Table 2 presents the physical properties obtained for the crude castor oil. The specific gravity value was found to be 0.956 approximately the same as the value (0.9587) obtained by (3), and agree with the ASTM standards propertiesof quality castor oil reported by (2) that is 0.962.

The viscosity of the crude castor oil was determined at 40°C and it was found to be 894.67 Cp. As can be seen the value of viscosity obtained for crude oil was significantly higher than that of ASTM standard reported in the literature (2). This may be attributed to the presence of some some impurities and components.

The chemical properties analysis shown in Table 3 indicates that the acid value of crude oil is 1.466mgKOH/100gm of oil. This result is slightly greater than the values of 1.231mg NaOH/g and 1.148 mg NaOH/g of Oil obtained by (2)and (3), however, it falls within the standard range of 0.40-4.0 specified in literature.

Table 3 shows the results for the saponification value of the crude and refined oil that were found to be 184.59mgKOH/g of oil. This result agree with the results of 185.83 mgKOH/g obtained by (3), and slightly different from the value of 179.33mg KOH/g and 180± 0.770 mg KOH/g oil obtained by( (3)and (1) respectively. However, all of them are highly comparable with ASTM specification for quality castor oil.

The free fatty acid was determined to be 0.733%. This can be used to check the level of oxidative deterioration of the oil by enzymatic or chemical oxidation. This value falls within the range of 0.00 -3.00% for free fatty acid of oil. However, the quality of the oil can be improved by refining so as to be used for industrial applications.

## CONCLUSION

It is concluded that sulphated castor oil is suitable as a fatliquoring of all types of leather. The raw material is available, cheap, toxic to human and nonedible.

## RECOMMENDATIONS

It is recommended to make leathers that can fatliquored with sulphated oil produced.

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# APPLICATION OF FATLIQUOR PREPARED FROM SUDANESE CASTOR OIL IN LEATHER FATLIQUORING PROCESS

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## ABSTRACT

Fatliquors are added to the leather during fatliquoring process to separate leather fibers from each other and to impart softness, flexibility and hand feeling. Fatliquors are mainly emulsifying mixtures, prepared by introduction of phosphate, sulphonate, and sulphiteqroups etc. into the structure of oils and fats or by addition of surfactants to the composition of fatliquoringagent.Sulphation is one of the common methods used to prepare fatliquor emulsion where sulphuric acid is used. In the present study, castor oil extracted from Sudanese castor beans in the previous work was used to prepare fatliqour by sulphation process. The physicochemical properties of the prepared fatliquor were tested. The fatliquor was applied to the bovine leathers in the fatliqouring process and the strength properties of the resultant crust leather were assessed through variousphysical and chemical tests.The results obtained revealed that Sudanese castor oil can effectively be used to prepare fatliquor to be used to lubricate leather in fatliquoring process.

**KEYWORDS**: Fatliquor, Sulphation, Sudanese castor oil.

## I. INTRODUCTION

The castor plant (Ricinuscommunis L.), belonging to the family Euphorbiaceae, and grows mostly in tropical and subtropical areas between latitudes 40˚ South and 52˚ North. It grows naturally over a wide range of geographical regions and may be cultivated under a variety of physical and climatic regions. (1).

Castor oil is a nonedible oil crop, pale amber viscous liquid and non-drying oil with mild or no odor or taste. Its propertied differ depending on the geographical location in which the plant is grown and the agricultural modifications which have been made during growth. (2)

Like any other vegetable oils it contains triglycerides, which chemically is a glycerol molecule with each of its three hydroxyl group esterified with a long clown fatty acid. Its major fatty acid is the unsaturated, hydroxylated 12-hydroxy, 9-octadecenoic acid, known familiarly as Ricinoleic acid. The fatty acid composition of a typical castor oil contains about 87% - 90 % of ricinoleic acid. (3)

It is reported that castor beans contains about 30-35% and can be extracted either by mechanical pressing or solvent extraction or combination of the two. Mechanical pressing only recovers about 45% of oil from the beans and the remainder in the cake can be recovered by solvent extraction using heptane, hexane or petroleum ethers .Actual yield depends on particular seed variety, geographical origin/climatic conditions, and on the oil extraction methods used(4).

Castor oil and its derivatives find outlet in industries and pharmaceuticals because of the ricinoleic acid, which predominates to about 89%, and is unusual because of the fact that it has a hydroxyl functional group on the twelfth carbon. The functional group causes ricinoleic acid to be unusually polar, and also allows chemical derivation that is not practical with most other seed oils. These characteristics, together with the oil's biodegradable and ecofriendly nature and its being a renewable resource, explain its limitless potentials(5)

Thus, the oil can be used in the production of vanishes, lacquers, protective coatings, lubricants, soaps, cosmetics, paints, inks, and it is a primary raw material for the production of nylon and other synthetic resins and fibers and a basic ingredient in racing motor oil for high-performance automobile motorcycle engines (6)

As we know, physical-mechanical properties and sensory properties are two important parameters for evaluating the application performance of leather and leather products. Softness is especially one of the most important physical properties to be taken into consideration when assessing the quality of light leathers (7).

In order to produce soft leather, after tanning the leather is processed through a fatliquoring step, which is designed to introduce oils and fats into the leather matrix preventing the adhesion of fibers. Fatliquoring is one of the critical steps for garment and upholstery leather manufacturing, which makes the leather soft and has a pleasant feel. The physical characteristics of the leather, as well as comfort properties of the leather, depend on fatliquoring(8).

Oil in water emulsions known as fatliquors are used to lubricate tanned leather fibres to get softness and also to improve the strength properties. An emulsion is a fine dispersion of one liquid in another liquid. Sulphation is one of the common methods followed to prepare fatliquor emulsion where sulphuric acid is used. In the conventional process, emulsifying agent is also added to increase the stability of oil in water emulsions. The emulsifying agents generally used are chemicals or metal soaps (9).

Fatliquor may be anionic, cationic or non-ionic. Anionic fatliquors are commonly employed for fat binding with chrome-tanned leather, which is cationically charged. Anionic fatliquors are commonly prepared by sulphation, sulphonation or bi-sulphitation of oils/fats(9).

Depending upon the source of the oils/fats used, the fatliquor can be classified as vegetable, synthetic and semi synthetic. Generally, castor oil is used as a source for vegetable based fatliquors. The synthetic fatliquors are usually obtained by sulphochlorination of C10 - C20 fractions obtained through the Fischer-Tropsch method of paraffin synthesis or from the petroleum industry. Semi-synthetic fatliquors are prepared from both the vegetable and synthetic sources (9).

Any fat or oil can be used as raw material for making a fatliquoringagent, the commonly available oils are those of cod oil, castor oil, neatsfoot oil, rapeseed oil, palm oil and sperm oil. All these oils have similar chemical structure of triglycerides. Castor oil is suitable as a basic raw material for the preparation of fatliquor due to hydroxyl functionality on ricinoleic acid (10).

Fatliquor affects the physical properties of the leather and makes it more flexible and softer (11).

The main characteristics of fatliquored leathers are feel, softness and a certain degree of water repellency. Physical properties such as tear resistance, break, and tensile strength as well as comfort properties of leathers depend on fatliquoring (12).

The objectives of the present work were to investigate the possibility of producing quality fatliqouring agent from abundant locally grown castor plant. Also, to assess the physical properties of the leathers produced after the application of the prepared fatliqour.

## Materials and methods

Castor oil was obtained from castor beans by means of solvent extraction. The chemical used for preparation of fatliquor such as Sulphuric acid, sodium chloride and sodium hydroxide were analytical grade. The chemical used in retanning process were commercial grade. Full grain chrome tanned goat skins were obtained from a local tannery.

### Preparation of fatliquor from castor oil

A concentrated sulphuric acid (98%) was added drop wise to 500 gm of castor oil with a constant stirring at 18-20°C. Thesulphation process was carried out slowly for about 3hrs. A saturated sodium chloride solution was added to the resultant products and mixed with them.The mixture was then kept in a separating funnel overnight to separate the layers. The upper layer was neutralized to pH 5.0 by adding 30% sodium hydroxide solution to produce the fatliquor.

**Analysis of fatliquor**

The sulphated castor oil was subjected to physical and chemical tests. The tests were carried out in Central Laboratory for Technical Services and calibration (CLTSC), by using NIST traceable reference equipment and materials in accordance with ISO/IEC 17025:2005 requirements and the test methods referenced below meets ISO/IEC 17025:2005 and accreditation bodies. Thetests were carried out at 20°C and 60% relative humidity.

The uncertainties values are calculated according to the standard uncertainty by coverage factor (k = 2) at 95% confidence level according to ISO 17025:2005 and accreditation body requirements as describing in the work instruction CLTSC/Work 1./5.4.1. The tests results were presented in Table 3.1

## II. RETANNINGAND FATLIQUORING PROCESS

Full grain chrome tanned bovine leathers of fairly similar size and free from physical defectsleatherswere wet back, retanned, fatliquored and converted into crust leather using the recipe presented in**Table 2.1**. The prepared sulphated castor oil was used for experimental leathers and an imported fatliquor was used for control leathers.The process was carried out using an experimental stainless steel drum.

*Table 2.1 Formulation of the retanning processes for experimental and control leathers*

| Process/products | % * | Time (min) | pH | Remarks |
|---|---|---|---|---|
| Wetting back | | | | |
| water | 200 | | | |
| Ps/A (wetting agent) | 0.3 | 30 | | |
| Formic acid | 0.3 | | | |
| Drain | | | | |
| Re-chroming | | | | |
| Water | 200 | | | |
| chrome | 2 | | | |
| Leave O/N, run 10′ morning, drain, wash | | | | |
| Neutralization | | | | |
| Water | 150 | | | |
| KNB( Syntan) | 1 | 30 | | |
| Sod-formate | 2 | | | |
| Sod- bicarbonate | 1 | 60 | 5 | |
| Drain, wash | | | | |
| Retanning | | | | |
| Water | 200 | | | |
| Mimosa | 3 | | | |
| DD7(syntan) | 3 | 40 | | |
| MTS (syntan) | 3 | | | |
| Coraline 6 | 1.5 | 10 | | |
| kw | 1.5 | 30 | | |
| PN3 | 1.5 | | | |
| Fatliquoring | | | | |
| MR | 2 | 30 | | |
| Filler | 3 | | | |
| Fatliqour | 6 | 45 | | |
| Formic acid | 1.5 | 40 | | |
| Drain, wash, pile O/N, toggle dry, trim and stake<br>*Percentages are based on wet blue weight. | | | | |

## III. PHYSICAL AND CHEMICAL TESTS OF THE CRUST LEATHERS

Physical and Chemical tests for the crust leathers were carried out by the Quality Control and Assurance Laboratory of the National Leather Technology Center (NLTC).

Thickness, tensile strength, tear strength, distention at grain crack and flexibilitytests of both experimental and control crust leathers were performed according to the standard methods (IUP). The readings averagesfor each test were presented in **Table 3.2**

Moisture, oil & fats and total ash contents of both experimental and control crust leathers were determined according to the official methods of analysis of (SLTC 1996). The readings averagesfor each test were shown in **Table 3.3.**

## IV. ANALYSIS OF SULPHATED CASTOR OIL

**Table 3.1**presents the physicochemical properties obtained for the sulphated castor oil.

***Table3.1 Physicochemical properties of sulphated castor oil***

| No. | Test | Test method No. | Result |
|---|---|---|---|
| 1 | Acid value (mgKOH/100gm) | AOAC | 0.82 |
| 2 | Free fatty acid (%) | AOAC | 0.414 |
| 3 | Specific Gravity (g/cm3) | AOAC2000 | 0.95714 |
| 4 | Viscosity@40°C (cps) | Cock and van(1966) | 2.43 |
| 5 | Saponification (Mg/g) | BS | 143.5 |

It is observed that the acid value of crude castor oil which was indicated in the previous work as 1.466mgKOH/100gm () decreased after sulphation to be 0.82 mgKOH/100gm however, both of them fall within the standard range of quality castor oil.

The free fatty acid of the sulphated castor oil was determined to be 0.414%. This value falls within the range of 0.00 -3.00% for free fatty acid of oil.

From the table the specific gravity value was found to be 0.95714 approximately the same as the valuereported by(11) for sulphated castoroil and agree with the ASTM standards properties of quality castor oil reported by (13) .

The viscosity of the sulphated castor oil was determined at 40°C and it was found to be 2.43 cps. The value of viscosity obtained is significantly higher than that of ASTM standard. This may be attributed to the presence of some impurities and components.

The value obtained for the saponification value of the prepared castor oil was found to be 143.5mg/mg. this value is less than the value of 176 mg/mg reported by(11),for sulphated castor oil. Andit is also less than the value reported by the author(14) for the raw castor oil and it is non confirmative with ASTM standards. Saponification value is inversely proportional to the molecular weight of the fatty acid present in oil.

## V. STRENGTH PROPERTIES

The values for physical tests for experimental and control leathers were presented in Table 3.2 Values for tensile strength, elongation and tear strength are the average values of fourdeterminations; two are parallel and two are perpendicular to the backbone. Values for load at grain crack are the average of three separate determinations.

*Table 3.2: Physical Testing Results for Experimental and Control Crust Leathers*

| Test | Average Readings | |
|---|---|---|
| | Experimental | Control |
| Thickness (mm) | 2.1 | 2.0 |
| Tensile strength (kg/cm2) | 334.8 | 330.5 |
| Elongation at break % | 59 | 52 |
| Single hole tear strength (kg/cm) | 137 | 138 |
| Tongue tear strength (kg/cm) | 40.5 | 49 |
| Double hole tear strength (kg/cm) | 178.5 | 280 |
| Distention at grain crack (mm) | 13.3 | 12.0 |
| Flexibility | Pass | Fail |

From the table it was observed that the strength properties of experimental leatherwere comparable to the strength properties of the control leathers. The results obtained were found within the limits of standard specification. The results shows that the prepared fatliqoure has good lubrication properties and possesses good penetration power and emulsion stability.

The high value of tensile strength indicates the high strength of collagen fibers. The elongation at break indicating the softness, flexibility, strength and toughness of leather. The experimental leathers pass the flexibility test while the control leather fail. This shows the good lubrication and penetration properties of the prepared fatliquor.

## VI. CHEMICAL ANALYSIS

The values for chemical analysis of experiments and control crust leathers were presented in **Table 3.3**

*Table 3.3Chemical Analysis Results for Experimental and Control Crust Leathers*

| Test | Readings Average | |
|---|---|---|
| | Experimental | Control |
| Moisture content % | 5.97 | 6.9 |
| Fat and Oil % | 8.50 | 10.9 |
| Ash content % | 7.11 | 8.7 |

The chemical characteristics of the experimental and control leathers were found to be quite normal. The moisture, fat and ash content, in leather seems to have acceptable value for both the experimental and control leathers.

## VII. CONCLUSION AND RECOMMENDATION

The physical and chemical characteristics of leathers produced the prepared fatliquor are comparable to that of leathers produced using the imoported fatliquor.so that the locally produced fatliquor from Sudanese castor oil would be a substitute to imported fatliquor.It is recommended to pay attention to castor plant cultivation for its tremendous uses of castor oil in industrial applications.

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