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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

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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

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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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