

Sudan University of Science and Technology

College of Science

Laboratory Chemistry Department

B.SC Complementary Research:

Preparation of Poly-Anhydride from Azelaic Acid

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صدق الله العظيم

سورة المجادلة
الآية (11)

Dedications

To your Fathers,

Mathers,

Sisters,

And All who in your mind

Acknowledgement

First of all always we thank our God (Allah) to make are live these moment.

We are very grateful to my dear supervision Fethy Abase we would like to him indeed for his supervision and encouragement. Also we would like to thank indeed my dear friends for help me create.

We send my greeting to my department, faculty and my dear university “SUST” and all there who helped to achieve the Bsc degree.

Last say: My parents, sisters, friends, Land, we missed you very much .you have a special place in heart.

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Abstract

poly azelaic anhydride was prepared from ricinoleic acid that was extracted from castor oil using di ethyl ether with low percent 32.14% due to commercial oil. with standard one T.L.C was carried out to iusure the purity and identify of the product, also IR was carried out to identify the functional groups present in the final product.

الملخص :

تم تحضير بولي أنهيدرايد من حمض الأزيلايك الناتج من أكسدة حمض الريسنولييك الموجود في زيت لخروج والذي تم استخلاصه عن طريق استخدام مذيب عضوي ,ثم فصل حمض الريسنواليك الناتج في شكل مادة زيتية ,ثم جمع المستخلص وحسبت النسبة المئوية ووجد أنها تساوي %32.4 وبمقارنتها بالنسبة القياسية % 85-95تعتبر أقل نسبة لاستخدام زيت تجاري والطريقة المستخدمة في الاستخلاص غير دقيقة و تم التأكد من الناتج عن طريق كروماتوغرافيا الطبقة الرقيقة وقياس IR.

Chapter One

1. Introduction

1.1Caster plant

The castor oil plant (*Ricinus communis*) is a species of flowering plant in the spurge family, Euphorbiaceae. It belongs to a monotypic genus, *Ricinus*, and subtribe, Riciniinae (Euphorbiaceae (spurge) (2009-03-09)).

Its seed is the castor bean, which, despite its name, is not a true bean. Castor is indigenous to the southeastern Mediterranean Basin, Eastern Africa, and India, but is widespread throughout tropical regions (and widely grown elsewhere as an ornamental plant). Castor seed is the source of castor oil, which has a wide variety of uses. The seeds contain between 40% and 60% oil that is rich in triglycerides, mainly ricinolein. The seed contains ricin, a toxin, which is also present in lower concentrations throughout the plant. (Phillips, etal (1999)).

1.1.1Description



Figure (1) Female (top) and male flowers

The castor oil plant can vary greatly in its growth habit and appearance. The variability has been increased by breeders who have selected a range of cultivars for leaf and flower colours, and for oil production. It is a fast-growing, suckering perennial shrub that can reach the size of a small tree (around 12 metres or 39 feet), but it is not cold hardy. The glossy leaves are 15–45 centimetres (5.9–17.7 in) long, long-stalked, alternate and palmate with 5–12 deep lobes with coarsely toothed segments. In some varieties they start off dark reddish purple or bronze when young, gradually changing to a dark green, sometimes with a reddish tinge, as they mature. The leaves of some other varieties are green practically from the start, whereas in yet others a pigment masks the green colour of all the chlorophyll-bearing parts, leaves, stems and young fruit, so that they remain a dramatic purple-to-reddish-brown throughout the life of the plant. Plants with the dark leaves can be found growing next to those with green leaves, so there is most likely only a single gene controlling the production of the pigment in some varieties. The

stems (and the spherical, spiny seed capsules) also vary in pigmentation. The fruit capsules of some varieties are more showy than the flowers.



Figure (2) Leaf of a Castor oil plant

The flowers are borne in terminal panicle-like inflorescences of green or, in some varieties, shades of red monoecious flowers without petals. The male flowers are yellowish-green with prominent creamy stamens and are carried in ovoid spikes up to 15 centimetres (5.9 in) long; the female flowers, borne at the tips of the spikes, have prominent red stigmas (Christopher Brickell, ed (1996)).

The fruit is a spiny, greenish (to reddish-purple) capsule containing large, oval, shiny, bean-like, highly poisonous seeds with variable brownish mottling. Castor seeds have a warty appendage called the caruncle, which is a type of elaiosome. The caruncle promotes the dispersal of the seed by ants (myrmecochory).

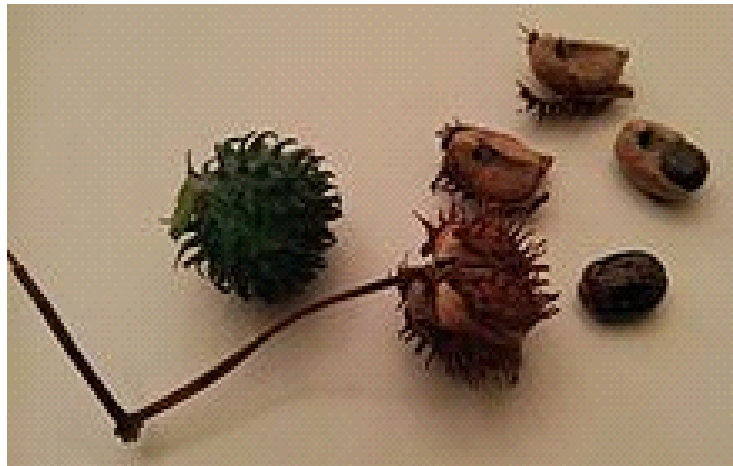


Figure (3) the green capsule dries and splits into three sections, forcibly ejecting seed

1.1.2 Nomenclature

The name *Ricinus* is a Latin word for tick; the seed is so named because it has markings and a bump at the end that resembles certain ticks. The common name "castor oil" 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). It has another common name, palm of Christ, or *Palma Christi*, that derives from castor oil's reputed ability to heal wounds and cure ailments.

1.1.3 Scientific classification

Kingdom	Plantae
unranked	Angiosperms
unranked	Eudicots
unranked	Rosids
Order	Malpighiales
Family	Euphorbiaceae
Subfamily	Acalyphoideae
Tribe	Acalypheae
Subtribe	Ricininae
Genus:	<i>Ricinus</i> L.
Species:	<i>R. communis</i>

1.1.4 Places cultivation

Castor plant (*Ricinus communis*) is cultivated around the world for its non-edible oilseed. Castor is an important non-edible oilseed crop and is grown especially in arid and semi arid region. It is originated in the tropical belt of both India and Africa. It is cultivated in 30 different countries on commercial scale, of which India, China, Brazil, USSR, Thailand Ethiopia and Philippines are major castor growing accounts about 88 per cent of the world's production. It is an annual crop and is grown by sowing the seeds in hot weather.



Figure (4) seeds

1.1.5 Ricin

The toxicity of raw castor beans is due to the presence of ricin. Although the lethal dose in adults is considered to be four to eight seeds, reports of actual poisoning are relatively rare. According to the 2007 edition of *Guinness World Records*, this plant is the most poisonous in the world. Despite this, suicides involving ingestion of castor beans are unheard of in countries like India where castor grows abundantly on the roadsides. The aversion to the use of the beans in suicide could be due to the painful and unpleasant symptoms of overdosing on ricin, which can include nausea, diarrhea, tachycardia, hypotension and seizures persisting for up to a week.

However, the poison can be extracted from castor by concentrating it with a fairly complicated process similar to that used for extracting cyanide from almonds .(*Toronto Star* Jun (1906)).

If ricin is ingested, symptoms may be delayed by up to 36 hours but commonly begin within 2–4 hours. These include a burning sensation in mouth and throat, abdominal pain, purging and bloody diarrhea. Within several days there is severe dehydration, a drop in blood pressure and a decrease in urine. Unless treated, death can be expected to occur within 3–5 days, however in most cases a full recovery can be made. Poisoning occurs when animals, including humans, ingest broken seeds or break the seed by chewing: intact seeds may pass through the digestive tract without releasing the toxin. (Soto-Blanco B, et al June (2002)) .

The toxin provides the castor oil plant with some degree of natural protection from insect pests such as aphids. Ricin has been investigated for its potential use as an insecticide. The castor oil plant is also the source for undecylenic acid, a natural fungicide. Commercially available cold-pressed castor oil is not toxic to humans in normal doses, either internal or externally (Irwin R March (1982)).

1.1.6 Uses of castor oil

1.1.6.1 Food and preservative

In the food industry, castor oil (food grade) is used in food additives flavorings, candy (*e.g.*, Polyglycerol polyricinoleate or PGPR in chocolate), (Wilson R et al (1998)).

As a mold inhibitor, and in packaging. Polyoxyethylated castor oil (*e.g.*, Kolliphor EL) is also used in the food industries. In India, Pakistan, Nepal and Bangladesh, food grains are preserved by applying castor oil. It stops rice, wheat, and pulses from rotting. For example the legume Toor dal is commonly available coated in oil for extended storage (Busso C et al March (2004)).

1.1.6.2 Medicine

The United States Food and Drug Administration (FDA) has categorized castor oil as "generally recognized as safe and effective" (GRASE) for over-the-counter use as a laxative with its major site of action the small intestine where it is digested into Ricinoleic acid. Therapeutically, modern drugs are rarely given in a pure chemical state, so most active ingredients are combined with excipients or additives. Castor oil, or a castor oil derivative such as Kolliphor EL (polyethoxylated castor oil, a nonionic surfactant), is added to many modern drugs, including:

- Miconazole, an antifungal agent;(Marmion LC et al September (1976)).
- Paclitaxel, a mitotic inhibitor used in cancer chemotherapy;(Micha JP et al February (2006)).
- Sandimmune (cyclosporine injection, USP), an immunosuppressant drug widely used in connection with organ transplant to reduce the activity of the patient's immune system.
- Nelfinavir mesylate, an HIV protease inhibitor;(Zhang KE et al April (2001)).
- Saperconazole, a triazole antifungal agent .
- Tacrolimus, an immunosuppressive drug (contains HCO-60, polyoxyl 60 hydrogenated castor oil)ᶦ

- Xenaderm ointment, a topical treatment for skin ulcers, is a combination of Balsam of Peru, castor oil, and trypsin; (San Antonio, TX, (2002)).
- Aci-Jel (composed of ricinoleic acid from castor oil, with acetic acid and oxyquinoline) is used to maintain the acidity of the vagina

1.1.6.2.1 Alternative medicinal use

In naturopathy castor oil has been promoted as a treatment for a variety of human health conditions. The claim has been made that applying it to the skin can help cure cancer. However, according to the American Cancer Society, "available scientific evidence does not support claims that castor oil on the skin cures cancer or any other disease."

1.1.6.3 Polyurethane

Castor oil can be used as bio-based polyol in the polyurethane industry. The average functionality (number of hydroxyl groups per triglyceride molecule) of castor oil is 2.7, so it is widely used as rigid polyol and coating

1.1.6.4 Industry

Castor oil has numerous applications in transportation, cosmetics and pharmaceutical, and manufacturing industries, for example: adhesives, brake fluids, caulks, dyes, electrical liquid dielectrics, humectants Nylon 11 plastics, hydraulic fluids, inks, lacquers, leather treatments lubricating greases, machining oils, paints, pigments, polyurethane adhesives, refrigeration lubricants, rubbers, sealants, textiles, washing powders, and waxes. Since it has a relatively high dielectric constant (4.7), highly refined and dried castor oil is sometimes used as a dielectric fluid within high performance high voltage capacitors.

1.1.6.5 Lubrication

Vegetable oils, due to their good lubricity and biodegradability are attractive alternatives to petroleum-derived lubricants, but oxidative stability and low temperature performance limit their widespread use. (McGuire, Nancy (2004)).

Castor oil has better low temperature viscosity properties and high temperature lubrication than most vegetable oils, making it useful as a lubricant in jet, diesel, and race car engines the viscosity of castor oil at 10°C is 2,420 centipoise. (Brady, etal (1997)).

However, castor oil tends to form gums in a short time, and its use is therefore restricted to engines that are regularly rebuilt, such as race engines. Biodegradability results in decreased persistence in the environment (relative to petroleum-based lubricants) in case of an accidental release. The lubricants company Castrol took its name from castor oil. Castor oil is the preferred lubricant for bicycle pumps, most likely because it does not dissolve natural rubber seals. (Jules (2000)).

1.1.6.6 Biodiesel

Castor oil, like currently less expensive vegetable oils, can be used as feedstock in the production of biodiesel. The resulting fuel is superior for cold winters, due to its exceptionally low cloud and pour points. (Carmen Leonor Barajas Forero, (2004-10-12)).

Initiatives to grow more castor for energy production, in preference to other oil crops, are motivated by social considerations. Tropical subsistence farmers would gain a cash crop. (Elizabeth Johnson (2004)).

1.2 Castor oil

Castor oil is a vegetable oil obtained by pressing the seeds of the Castor plant (*Ricinus communis*). 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). Castor oil is famous as a source of ricinoleic acid, a monounsaturated, 18-carbon fatty acid. Among fatty acids, ricinoleic acid is unusual in that it has a hydroxyl functional group on the 12th carbon. This functional group causes ricinoleic acid (and castor oil) to be more polar than most fats. The chemical reactivity of the alcohol group also allows chemical derivatization that is not possible with most other seed oils. Because of its ricinoleic acid content, castor oil is a valuable chemical in feedstocks, commanding a higher price than other seed oils.

1.2.1 components of castor oil

Average composition of seed oil /fatty acid chains

Acid name	Average Percentage Range		
Ricinoleic acid	95	to	85%
Oleic acid	6	to	2%
Linoleic acid	5	to	1%
Linolenic acid	1		0.5%
Stearic acid	1	to	0.5%
Palmitic acid	1	to	0.5%
Dihydroxystearic acid	0.5	to	0.3%
Others	0.5	to	0.2%

1.2.2 Properties of castor oil

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/m³. It is a triglyceride in which approximately 90 percent of fatty acid chains are ricinoleate. Oleate and linoleates are the other significant component.

1.2.3 Uses of Components of castor oil

1.2.3.1 Ricinoleic acid

Ricinoleic acid, formally called 12-hydroxy-9-cis-octadecenoic acid is a fatty acid. It is an unsaturated omega-9 fatty acid. It is a major component of the seed oil obtained from mature Castor plant (*Ricinus communis* L., Euphorbiaceae) seeds or in sclerotium of ergot (*Claviceps*

purpurea Tul, Clavicipitaceae). About 90% of the fatty acid content in castor oil is the triglyceride formed from ricinoleic acid.

1.2.3.1.1 Uses

Ricinoleic acid is manufactured for industries by saponification or fractional distillation hydrolyzed castor oil (James AT,etal May (1965)).

The zinc salt is used in personal care products such as deodorants (Tom's of Maine). The first attempts to prepare ricinoleic acid were made by Friedrich Krafft in 1888 Biological activities (Rider, T.H. November(1931)).

Ricinoleic acid exerts analgesic and anti-inflammatory effects.(Vieira C, etal (2000)).

Ricinoleic acid specifically activates the EP3 prostanoid receptor for prostaglandin E2 Ricinoleic acid acts as a specific algicide for the control of blue-green algae. (Diener M etal (2012)).

1.2.3.2 Oleic acid

Oleic acid is a fatty acid that occurs naturally in various animal and vegetable fats and oils. It is an odorless, colourless oil, although commercial samples may be yellowish. In chemical terms, oleic acid is classified as a monounsaturated 99omega-9 fatty acid, abbreviated with a lipid number of 18:1 2%*cis*-9. It has the formula $\text{CH}_3(\text{CH}_2)_7\text{CH}=\text{CH}(\text{CH}_2)_7\text{COOH}$. The term "oleic" means related to, or derived from, oil or olive, the oil that is predominantly composed of oleic acid. Oleic acid (in triglyceride form) is included in normal human diet as part of animal fats and vegetable oil(Thomas, Alfred(2000))

1.2.3.2.1 Uses

Oleic acid as its sodium salt is a major component of soap as an emulsifying agent. It is also used as emollient(Carrasco, F (2009)).

Small amounts of oleic acid are used as an excipient in pharmaceuticals, oleic acid is used as an emulsifying or solubilizing agent in aerosol products. (Carrasco, F. (2009)).

Oleic acid is also used to induce lung damage in certain types of animals, for the purpose of testing new drugs and other means to treat lung diseases. Specifically in sheep, intravenous administration of oleic acid causes acute lung injury with corresponding pulmonary edema. (Julien, M;etal (1986)).

This sort of research has been of particular benefit to premature newborns, for whom treatment for underdeveloped lungs (and associated complications) often is a matter of life and death. Oleic acid is used as a soldering flux in stained glass work for joining lead came. (Duncan, Alastair (2003)).

1.2.3.3 Linoleic acid

Linoleic acid (LA) is a polyunsaturated 66omega-6 fatty acid. It is a colorless liquid at room temperature. In physiological literature, it has a lipid number of 18:2 *cis,cis*-9,12.

Chemically, linoleic acid is a carboxylic acid with an 18-carbon chain and two *cis* double bonds; with the first double bond located at the sixth carbon from the methyl end.(David J. Anneken, etal (2006)).

Linoleic acid belongs to one of the two families of essential fatty acids, which means that the human body cannot synthesize it from other food components .The word "linoleic" comes from the Greek word *linon* (flax). *Oleic* means "of, relating to, or derived from oil of olive" or "of or relating to oleic acid" because saturating the omega-6 double bond produces oleic acid.(Miller, E. (1930)).

1.2.3.3.1 Industrial uses

Linoleic acid is used in making quick-drying oils, which are useful in oil paints and varnishes. These applications exploit the easy reaction of the linoleic acid with oxygen in air, which leads to crosslinking and formation of a stable film. Reduction of linoleic acid yields linoleyl alcohol. Linoleic acid is a surfactant with a critical micel concentration of 1.5×10^{-4} M @ pH 7.5. Linoleic acid has become increasingly popular in the beauty products industry because of its beneficial properties on the skin. Research points to linoleic acid's anti-inflammatory, acne reductive, and moisture retentive properties when applied topically on the skin.(Schulz, etal (1993)), (Letawe, C;etal March (1998)) and(Darmstadt, etal (2002)).

1.2.3.3.2 Use in research

Linoleic acid can be used to show the antioxidant effect of natural phenols. Experiments on linoleic acid subjected to 2222,(%22222,2'-azobis (2-amidinopropane) dihydrochloride-induced oxidation with different combinations of phenolics show that binary mixtures can lead to either a synergetic antioxidant effect or to an antagonistic effect. (Peyrat-Maillard. (2003)).

Linoleic acid may be linked to obesity by promoting overeating and damaging the arcuate nucleus in the brain's hypothalamus.(Janet Raloff (2012)).

1.2.3.4 Stearic acid

Stearic acid is the saturated fatty acid with an 18-carbon chain and has the IUPAC name octadecanoic acid. It is a waxy solid, and its chemical formula is $\text{CH}_3(\text{CH}_2)_{16}\text{CO}_2\text{H}$. Its name comes from the Greek word *στέαρ* "*stéar*", which means tallow. The salts and esters of stearic acid are called stearates. Stearic acid is one of the most common saturated fatty acids found in nature following palmitic acid. (Gunstone, F. D, etal (2007)).

1.2.3.4.1 Uses

Stearic acid is mainly used in the production of detergents, soaps, and cosmetics such as shampoos and shaving cream products. Soaps are not made directly from stearic acid, but indirectly by saponification of triglycerides consisting of stearic acid esters. Esters of stearic acid with ethylene glycol; glycol stearate and glycol distearate, are used to produce a pearly effect in shampoos, soaps, and other cosmetic products. They are added to the product in molten form and allowed to crystallize under controlled conditions. Detergents are obtained from amides and quaternary alkylammonium derivatives of stearic acid.

1.2.3.4.1.1 Lubricants, softening and release agents

In view of the soft texture of the sodium salt, which is the main component of soap, other salts are also useful for their lubricating properties. Lithium stearate is an important component

of grease. The stearate salts of zinc, calcium, cadmium, and lead are used to soften PVC. Stearic acid is used along with castor oil for preparing softeners in textile sizing. They are heated and mixed with caustic potash or caustic soda. Related salts are also commonly used as release agents, e.g. in the production of automobile tires.

1.2.4.1.2 Niche uses

Being inexpensively available and chemically benign, stearic acid finds many niche applications, for example, in making plaster castings from a plaster *piece mold* or *waste mold* and in making the mold from a shellacked clay original. In this use, powdered stearic acid is mixed in water and the suspension is brushed onto the surface to be parted after casting. This reacts with the calcium in the plaster to form a thin layer of calcium stearate, which functions as a release agent. When reacted with zinc it forms zinc stearate, which is used as a lubricant for playing cards (fanning powder) to ensure a smooth motion when fanning. In compressed confections, it is used as a lubricant to keep the tablet from sticking to the die.

Stearic acid is also used as a negative plate additive in the manufacture of lead-acid batteries. It is added at the rate of 0.6 g per kg of the oxide while preparing the paste (Lam et al (1998)).

It is believed to enhance the hydrophobicity of the negative plate, particularly during dry-charging process. It also reduces the extension of oxidation of the freshly formed lead (negative active material) when the plates are kept for drying in the open atmosphere after the process of tank formation. As a consequence, the charging time of a dry uncharged battery during initial filling and charging (IFC) is comparatively lower, as compared to a battery assembled with plates which do not contain stearic acid additive. Fatty acids are classic components of candle-making. Stearic acid is used along with simple sugar or corn syrup as a hardener in candies. Stearic acid is used to produce dietary supplements. In fireworks, stearic acid is often used to coat metal powders such as aluminium and iron. This prevents oxidation, allowing compositions to be stored for a longer period of time. Stearic acid is a common lubricant during injection molding and pressing of ceramic powders. (Tsenga et al (1999)).

It is also used as a mold release for foam latex that is baked in stone molds.

1.2.3.5 Palmitic acid

Palmitic acid, or hexadecanoic acid in IUPAC nomenclature, is the most common fatty acid (saturated) found in animals, plants and microorganisms. (Gunstone, et al (2007)).

Its molecular formula is $(\text{CH}_3(\text{CH}_2)_{14}\text{COOH})$. As its name indicates, it is a major component of the oil from palm trees (palm oil, palm kernel, and palm kernel oil), but can also be found in meats, cheeses, butter, and dairy products. Palmitate is a term for the salts and esters of palmitic acid. The palmitate anion is the observed form of palmitic acid at physiologic pH (7.4). Aluminium salts of palmitic acid and naphthenic acid were combined during World War II to produce napalm. The word "napalm" is derived from the words naphthenic acid and palmitic acid.

1.2.3.5 .1Uses

Palmitic acid is mainly used to produce soaps, cosmetics, and release agents. These applications utilize sodium palmitate, which is commonly obtained by saponification of palm oil. To this end, palm oil, rendered from the coconut palm nut, is treated with sodium hydroxide (in the form of caustic soda or lye), which causes hydrolysis of the ester groups. This procedure affords glycerol and sodium palmitate. Because it is inexpensive and adds texture to processed foods (convenience food), palmitic acid and its sodium salt find wide use including foodstuffs. Sodium palmitate is permitted as a natural additive in organic products. Hydrogenation of palmitic acid yields cetyl alcohol, which is used to produce detergents and cosmetics. Recently, a long-acting antipsychotic medication, paliperidone palmitate (marketed as INVEGA Sustenna), used in the treatment of schizophrenia, has been synthesized using the oily palmitate ester as a long-acting release carrier medium when injected intramuscularly. The underlying method of drug delivery is similar to that used with decanoic acid to deliver long-acting depot medication, in particular, neuroleptics such as haloperidol decanoate.

1.3 carboxylic acids

An acid (from the Latin *acidus/acēre* meaning *sour*) is a chemical substance whose aqueous solutions are characterized by a sour taste, the ability to turn blue litmus red, and the ability to react with bases and certain metals (like calcium) to form salts. Aqueous solutions of acids have a pH of less than 7. A lower pH means a higher acidity, and thus a higher concentration of hydrogen ions in the solution. Chemicals or substances having the property of an acid are said to be acidic. Common examples of acids include hydrochloric acid (a solution of hydrogen chloride which is found in gastric acid in the stomach and activates digestive enzymes), acetic acid (vinegar is a dilute solution of this liquid), sulfuric acid (used in car batteries), and tartaric acid (a solid used in baking). As these examples show, acids can be solutions or pure substances, and can be derived from solids, liquids, or gases. Strong acids and some concentrated weak acids are corrosive, but there are exceptions such as carboranes and boric acid. There are three common definitions for acids: the Arrhenius definition, the Brønsted-Lowry definition, and the Lewis definition. The Arrhenius definition defines acids as substances which increase the concentration of hydrogen ions (H^+), or more accurately, hydronium ions (H_3O^+), when dissolved in water. The Brønsted-Lowry definition is an expansion: an acid is a substance which can act as a proton donor. By this definition, any compound which can easily be deprotonated can be considered an acid. Examples include alcohols and amines which contain O-H or N-H fragments. A Lewis acid is a substance that can accept a pair of electrons to form a covalent bond. Examples of Lewis acids include all metal cations, and electron-deficient molecules such as boron trifluoride and aluminium trichloride.

1.3.1 Nomenclature

In the classical naming system, acids are named according to their anions. That ionic suffix is dropped and replaced with a new suffix (and sometimes prefix), according to the table below. For example, HCl has chloride as its anion, so the -ide suffix makes it take the form hydrochloric acid. In the IUPAC naming system, "aqueous" is simply added to the name of the ionic compound. Thus, for hydrogen chloride, the IUPAC name would be aqueous hydrogen chloride. The prefix "hydro-" is added only if the acid is made up of just hydrogen and one other element.

1.3.1.1 Acid strength

- Acid strength

The strength of an acid refers to its ability or tendency to lose a proton. A strong acid is one that completely dissociates in water; in other words, one mole of a strong acid HA dissolves in water yielding one mole of H^+ and one mole of the conjugate base, A^- , and none of the protonated acid HA. In contrast, a weak acid only partially dissociates and at equilibrium both the acid and the conjugate base are in solution. Examples of strong acids are hydrochloric acid (HCl), hydroiodic acid (HI), hydrobromic acid (HBr), perchloric acid ($HClO_4$), nitric acid (HNO_3) and sulfuric acid (H_2SO_4). In water each of these essentially ionizes 100%. The stronger an acid is, the more easily it loses a proton, H^+ . Two key factors that contribute to the ease of deprotonation are the polarity of the H—A bond and the size of atom A, which determines the strength of the H—A bond. Acid strengths are also often discussed in terms of the stability of the conjugate base. Stronger acids have a larger K_a and a more negative pK_a than weaker acids.

Sulfonic acids, which are organic oxyacids, are a class of strong acids. A common example is toluenesulfonic acid (tosylic acid). Unlike sulfuric acid itself, sulfonic acids can be solids. In fact, polystyrene functionalized into polystyrene sulfonate is a solid strongly acidic plastic that is filterable.

Superacids are acids stronger than 100% sulfuric acid. Examples of superacids are fluoroantimonic acid, magic acid and perchloric acid. Superacids can permanently protonate water to give ionic, crystalline hydronium "salts". They can also quantitatively stabilize carbocations. While K_a measures the strength of an acid compound, the strength of an aqueous acid solution is measured by pH, which is an indication of the concentration of hydronium in the solution. The pH of a simple solution of an acid compound in water is determined by the dilution of the compound and the compound's K_a .

1.3.1.2 Weak acid–weak base equilibrium

- Henderson–Hasselbalch equation

In order for a protonated acid to lose a proton, the pH of the system must rise above the pK_a of the acid. The decreased concentration of H^+ in that basic solution shifts the equilibrium towards the conjugate base form (the deprotonated form of the acid). In lower-pH (more acidic) solutions, there is a high enough H^+ concentration in the solution to cause the acid to remain in its protonated form. Solutions of weak acids and salts of their conjugate bases form buffer solutions.

1.3.2 Applications of acids

There are numerous uses for acids. Acids are often used to remove rust and other corrosion from metals in a process known as pickling. They may be used as an electrolyte in a wet cell battery, such as sulfuric acid in a car battery. Strong acids, sulfuric acid in particular, are widely used in mineral processing. For example, phosphate minerals react with sulfuric acid to produce phosphoric acid for the production of phosphate fertilizers, and zinc is produced by dissolving zinc oxide into sulfuric acid, purifying the solution and electrowinning. In the chemical industry, acids react in neutralization reactions to produce salts. For example, nitric acid reacts with ammonia to produce ammonium nitrate, a fertilizer. Additionally, carboxylic acids can be esterified with alcohols, to produce esters.

Acids are used as additives to drinks and foods, as they alter their taste and serve as preservatives. Phosphoric acid, for example, is a component of cola drinks. Acetic acid is used in day-to-day life as vinegar. Carbonic acid is an important part of some cola drinks and soda. Citric acid is used as a preservative in sauces and pickles. Tartaric acid is an important component of some commonly used foods like unripened mangoes and tamarind. Natural fruits and vegetables also contain acids. Citric acid is present in oranges, lemon and other citrus fruits. Oxalic acid is present in tomatoes, spinach, and especially in carambola and rhubarb; rhubarb leaves and unripe carambolas are toxic because of high concentrations of oxalic acid. Ascorbic acid (Vitamin C) is an essential vitamin for the human body and is present in such foods as amla, lemon, citrus fruits, and guava. Certain acids are used as drugs. Acetylsalicylic acid (Aspirin) is used as a pain killer and for bringing down fevers. Acids play important roles in the human body. The hydrochloric acid present in the stomach aids in digestion by breaking down large and complex food molecules. Amino acids are required for synthesis of proteins required for growth and repair of body tissues. Fatty acids are also required for growth and repair of body tissues. Nucleic acids are important for the manufacturing of DNA and RNA and transmitting of traits to offspring through genes. Carbonic acid is important for maintenance of pH equilibrium in the body.

1.3.2.1 Acid catalysis

- **Acid catalysis**

Acids are used as catalysts in industrial and organic chemistry; for example, sulfuric acid is used in very large quantities in the alkylation process to produce gasoline. Strong acids, such as sulfuric, phosphoric and hydrochloric acids also effect dehydration and condensation reactions. In biochemistry, many enzymes employ acid catalysis.

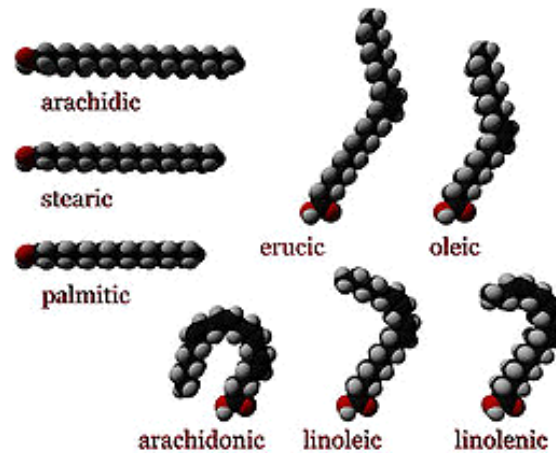
Dicarboxylic acids are organic compounds that contain two carboxylic acid functional groups. In molecular formulae for dicarboxylic acids, these groups are sometimes written as HOOC-R-COOH. Dicarboxylic acids are used to prepare copolymers such as polyamides and polyesters.

1.3.2.2 Fatty acid

In chemistry, and especially in biochemistry, a fatty acid is a carboxylic acid with a long aliphatic tail (chain), which is either saturated or unsaturated. Most naturally occurring fatty acids have a chain of an even number of carbon atoms, from 4 to 28. Fatty acids are usually derived from triglycerides or phospholipids. When they are not attached to other molecules, they

are known as "free" fatty acids. Fatty acids are important sources of fuel because, when metabolized, they yield large quantities of ATP. Many cell types can use either glucose or fatty acids for this purpose. In particular, heart and skeletal muscle prefer fatty acids. Despite long-standing assertions to the contrary, the brain can use fatty acids as a source of fuel in addition to glucose and ketone bodies. (Ebert, D etal (2003))and(Marin-Valencia etal 17 October (2012)).

1.3.2.3Types of fatty acids



Figure(5)Three-dimensional representations of several fatty acids

Fatty acids that have 2% carbon–carbon double bonds are known as unsaturated. Fatty acids without double bonds are known as saturated. They differ in length as well.

1.3.2.Length of free fatty acid chains

Fatty acid chains differ by length, often categorized as short to very long.

- Short-chain fatty acids (SCFA) are fatty acids with aliphatic tails of fewer than six carbons (i.e. butyric acid). (John Wiley etal(2013)).
- Medium-chain fatty acids (MCFA) are fatty acids with aliphatic tails of 6–12carbons, (Roth, Karl S (2013-12-19))
- which can form medium-chain triglycerides.
- Long-chain fatty acids (Beermann, C.; etal (2003)).
- Very long chain fatty acids (VLCFA) are fatty acids with aliphatic tails longer than 22 carbons

1.4 Unsaturated fat

An unsaturated fat is a fat or fatty acid in which there is at least one double bond within the fatty acid chain. A fatty acid chain is monounsaturated if it contains one double bond, and polyunsaturated if it contains more than one double bond. Where double bonds are formed, hydrogen atoms are eliminated. Thus, a saturated fat has no double bonds, has the maximum number of hydrogens bonded to the carbons, and therefore is "saturated" with hydrogen atoms. In cellular metabolism, unsaturated fat molecules contain somewhat less energy (i.e., fewer calories) than an equivalent amount of saturated fat. The greater the degree of unsaturation in a

fatty acid (i.e., the more double bonds in the fatty acid) the more vulnerable it is to lipid peroxidation (rancidity). Antioxidants can protect unsaturated fat from lipid peroxidation. Examples of unsaturated fats are palmitoleic acid, oleic acid, myristoleic acid, linoleic acid, and arachidonic acid.

1.4.1 Reactions of fatty acids

1.4.1.1 Acidity

Fatty acids do not show a great variation in their acidities, as indicated by their respective pK_a . Nonanoic acid, for example, has a pK_a of 4.96, being only slightly weaker than acetic acid (4.76). As the chain length increases, the solubility of the fatty acids in water decreases very rapidly, so that the longer-chain fatty acids have minimal effect on the pH of an aqueous solution. Even those fatty acids that are insoluble in water will dissolve in warm ethanol, and can be titrated with sodium hydroxide solution using phenolphthalein as an indicator to a pale-pink endpoint. This analysis is used to determine the free fatty acid content of fats; i.e., the proportion of the triglycerides that have been hydrolyzed.

1.4.1.2 Hydrogenation and hardening

Hydrogenation of unsaturated fatty acids is widely practiced to give saturated fatty acids, which are less prone toward rancidification. Since the saturated fatty acids are higher melting than the unsaturated relatives, the process is called hardening. This technology is used to convert vegetable oils into margarine. During partial hydrogenation, unsaturated fatty acids can be isomerized from *cis* to *trans* configuration.



Alkene plus hydrogen yields an alkane.

More forcing hydrogenation, i.e. using higher pressures of H_2 and higher temperatures, converts fatty acids into fatty alcohols. Fatty alcohols are, however, more easily produced from fatty acid esters. In the Varrentrapp reaction certain unsaturated fatty acids are cleaved in molten alkali, a reaction at one time of relevance to structure elucidation.

1.4.1.3 Ozonolysis

Unsaturated fatty acids are susceptible to degradation by ozone. This reaction is practiced in the production of azelaic acid $((\text{CH}_2)_7(\text{CO}_2\text{H})_2)$ from oleic acid. (Anneken *etal.* (2006)).

Chapter Two

2.Experimental and Results

2.1 Chemicals

- Potassium hydroxide

Mwt: 56.11

Batch No: 06093

Description: white deliques cent flakes

- Potassium permanganate

Mwt: 158.03

CAS No: 77722.64.

Assay: 98%

- Sulfuric acid:

Mwt: 98.08

Art: 748

- Thionyl chloride

M.w 116.99

Batch No .To220

Assay (min) 99%

- Cyclohexane

M.Wt 84.16

Batch No 0.9o932

Assay 99%

Freezing point 5.0-65 c0

-N,Ndiethyl aniline

M.w 149.25

Batch No o25652

Assay (Glc) 98.5%

Solvent

-di ethyl ether

Sr: No al2999

Batch No: p0142

Acidity: 0.01 mol N% W% V%

Alkalinity: 0.01 ml W% W/V

CAS No: 8032-32-4

-ethanol

2.2 Equipment

Beakers (100-400ml) - glass rod -conical flask (250ml) -graduated cylinder (50 ml) -funnel - stand -filter paper -separating funnel -Round bottom flask.

2.3 Instrument

Analytical balance - condensar - simble distillation balance - hot plate.

2.4 methode

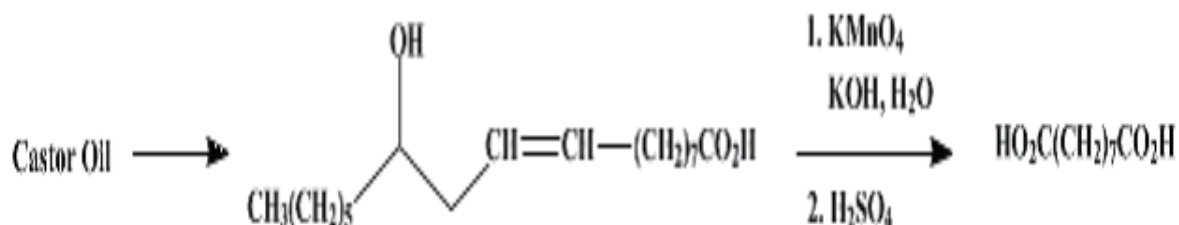
2.4.1 Preparation of ricinoleic acid from castor oil

56 g castor oil was filled in a 250 mL round bottom flask with magnetic stirbar and reflux condenser, containing a solution of 33.0 g KOH in 120 mL ethanol. The mixture is heated under stirring for 3 hours under reflux. The cooled mixture was poured in a 400 mL beaker containing 80 g ice. Then carefully, under strong stirring with a glass rod the solution of 30 mL conc. sulphuric acid in 60 mL water was added until clear acidic reaction; thereby the crude ricinoleic acid separates as oil. The phases are separated in the separating funnel; the aqueous phase was extracted with 40 mL di ethyl ether. Oil and ether extract were combined and the solvent was evaporated at a rotary evaporator. The ricinoleic acid remains as oily residue.

2.4.2 Oxidation of ricinoleic acid (from castor oil) with KMnO_4 To azelaic acid

8.03 g crude ricinoleic acid was dissolved in a 250 mL Erlenmeyer flask, containing a solution of 2.4 g KOH in 90 mL water. In conical flask equipped and internal thermometer, 21 g potassium permanganate was dissolved in 232 mL water at an internal temperature of about 35°C. Under strong stirring the alkaline solution in the Erlenmeyer flask was added at once to the ricinoleic acid in the threeneck flask. A reflux condenser is installed on the free opening of the flask and the mixture is stirred without further heating or cooling until the colour of permanganate has disappeared (30 minutes). For a better examination of the colour, a small sample of the solution is diluted in a test tube with water. The reflux condenser was replaced for a short time through an addition funnel with pressure balance. Under stirring a solution of 20 mL conc. sulfuric acid in 140 mL water was slowly added dropwise. In order to reach a better conglomeration and precipitation of the product. The crude product is dissolved in about 100 mL

hot water and filtered while hot. It was slowly cooled down to room temperature, again cooled in an ice bath, sucked off, then washed by 30 mL ice water and dried in the desiccator.



2.4.3 Preparation of acid chloride

The azelaic acid 8.65 g (was treated with thionyl chloride at 80 C. for 6 hours. The acid chloride was distilled off B.P. 108-110 C. /0.6 mm. yield 114 g. The acid chloride 9.35 m was dissolved in cyclo hexane 2.8 m and to this n. diethyl anilin 2.8 m was added at room temperature and the mix set aside for 48 hours in absence of moisture. Precipitated n. diethyl anilin hydrochloride was filtered off and cyclo hexane distilled off from the filtrate. A part of the residue was saponified; with alcoholic potash and then acidified after removal of alcohol when civetone-dicarboxylic acid was obtained as a precipitate. This was crystallised from benzene M.P. 114 C. Another portion of the residue was refluxed for 6 hours with aqueous alcohol when ethyl ester of civetone-dicarboxylic acid was obtained, M.P. 50 C.

Chapter Three

Results:

1-Ricinoleic acid Isolation :

Weight of the Beaker empty (g)	Weight of the Beaker is full(g)	Weight of precipitat (g)
50.32	68.32	18

The yield% 100

The% 100 = 32.14 %

2- Prepration of azelaic acid

Weight of the Beaker isempty (g)	Weight of the Beaker isfull(g)	Weight of precipitat (g)
1.02	11.44	10.42

-No mole of potassium permanganate = $Wt (g) / Mwt$

$$= 31.6 / 158.03 = 0.199 \text{ mol}$$

-No mole of ricinoleic acid = $12 / 298.3 = 0.0402 \text{ mole}$

-No mole of product = No mol of ricinoleic acid = 0.0402 mole

-Theoretical weight = No of mole * Mwt

$$= 0.0402 * 188.2 = 7.57 \text{ g}$$

% yeild = practical weight / theoretical weight

$$= 10.42 / 7.57 * \%100 = 137 \%$$

3- Analytics TLC

The system	The component	Number of component	Colour of component	Distance traveled by components	Distance traveled by solvent front
Aceton	Anhydride	1	Yellow	0.70	4.50
	Anhydride	1	Yellow	0.80	4.50

TLC-conditions:

Adsorbant: acetone, 4.9 x 7cm

Eluent: ethanol

Visualization: Iodine -reagent-TLC-solution. The spots appear as yellow marks after the plate was

dipped in the solution and dried by a slight heating with a hot-air dryer.

Reagent solution:

RF

RF = 0.15

RF = 0.17

4- IR spectrum of the pure product

(cm-1)	Assignment
2500-3300	2500-3300 O-H-valence, carboxylic acid
2962, 2872	C-H-valence, alkane, O-H
1735	C=O-valence, carboxylic acid
1800,1760	Anhydride

Discussion:

Castor oil is famous as a source of ricinoleic acid, a monounsaturated, 18-carbon fatty acid. Among fatty acids, ricinoleic acid is unusual in that it has a hydroxyl functional group on the 12th carbon. This functional group causes ricinoleic acid (and castor oil) to be more polar than most fats. The chemical reactivity of the alcohol group also allows chemical derivatization that is not possible with most other seed oils. Because of its ricinoleic acid content, castor oil is a valuable chemical in feedstocks, commanding a higher price than other seed oils.

Ricinoleic acid was extracted from commercial castor oil using diethyl ether as solvent then the extract was treated with potassium hydroxide the filtrate was treated with sulphuric acid to regenerate the acid the percentage yield was calculated the acid was treated with potassium permanganate to obtain azelaic acid which was converted to acid chloride using thionyl chloride the product was treated with azelaic acid to produce the poly anhydride with standard one T.L.C was carried out to insure the purity and identify of the product, also IR was carried out to identify the functional groups present in the final product. The spectrum of the sample is similar to the standard.

Recommendations

The ricinoleic acid was extracted from castor oil and converted to azelaic acid which converted to poly azelaic anhydride the following point should be done .

1-commercial castor oil gives small product so that pure sample is better to be used.

2- more efficient method should be used

3- more characterization procedure such as HNMR ,M.S should be used

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