



WOLKITE UNIVERSITY

COLLEGE OF ENGINEERING AND TECHNOLOGY

DEPARTMENT OF CHEMICAL ENGINEERING

TITLE: PRODUCTION OF BIOLUBRICANT FROM CASTOR OIL

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DECLARATION

We are fifth year student from college of engineering and technology department of chemical engineering and also it took us two months to complete this research. We declared that this document is our independent work, all written material presented in this document containing things are done by our effort except suggestion of our advisor and we used different source.

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ABSTRACT

Bio-lubricant is renewable substitute for petroleum based lubricant, which is non toxic, biodegradable, and renewable source. If reacting castor oil with methanol or ethanol with the help of NaOH or KOH as a catalyst in the esterification process to produce mono ester that are termed as fatty acid methyl ester. That process affected by mode of reaction, type of alcohol, ratio of alcohol to oil and catalyst. After production to know and characterize the physicochemical property (flash point, pour point, viscosity, iodine value) of bio-lubricant is necessary. The production of bio-lubricant from castor oil in order to utilize renewable resource and solve environmental issue related to petroleum lubricant. When compared with petroleum based lubricant, it has low toxicity, high viscosity, highly biodegradable and cost saving on disposal storage. This paper carried out experimental study, through extraction and characterization of both refined castor oil and bio-lubricant. From parameters extraction time and temperature affects the yield of oil, when time of extraction increased result to increase oil yield and small value of temperature also gain small amount of oil, so maximum yield of castor oil obtained at 78°C for ethanol solvent and 65°C for methanol solvent.

Keywords :Castor seed, Castor refined oil, Degumming, bio lubricant, Transesterification, Methyl ester, Trimethylolpropane ester.

ACRONYM OR ABBREVIATIONS

ASTM	American Society for Testing and Material
AV	Acid value
C	Carbon
COFAME	Castor Oil Fatty Acid Methyl Ester
Na OH	Sodium hydroxide
EN	European Standard
EP	Extreme pressure
FAME	Fatty Acid Methyl Ester
FFA	Free Fatty Acid
H C	Hydrochloric acid
ISOVG	International Standard Organization Viscosity Grade
MG	Mono glycerine
SCCO2	Supercritical CO2
SCF	Supercritical Fluid
SLS	Sodium lauryl sulphate
TAN	Total acid number
TG	Triglyceride
TPM	Trim-ethyl propane
VI	Viscosity index

CHAPTER ONE

1 INTRODUCTION

1.1 Background

Bio-lubricant Formulations is from vegetable based oils together with corresponding additives which has final composition of 60–99% of base oil and the remaining as additive, depending on the desired performance[7]. Recently, increasing attention to environmental issues has driven the lubricant industry toward eco-friendly products from renewable sources. The use of biodegradable and environmentally accepted lubricants from vegetable oil has increased over the past 25 years. However, millions of tons of lubricants are dumped into the environment through leakage and careless disposal. Some of these wastes are resistant to bio-degradation and are threats to the environment[7].

Bio lubricant is a renewable substitute for petroleum based lubricant which is made from nontoxic, biodegradable, and renewable sources such as refined and used vegetable oils. The unsaturated free fatty acid which is defined as the ratio and position of carbon-carbon double bond, one, two and three double bonds of carbon chain is named as a oleic, linoleic, and linolenic fatty acid components respectively. Although vegetable oils possess many desirable characteristics, currently they are not widely used as lubricant base oils. Largely this is due to undesirable physical properties of most vegetable oils, which include both a high melting point and insufficient thermal oxidative stability[12].

Chemical modifications may improve the thermal, oxidative and hydrolytic stabilities of the vegetable oils. Transesterification, modifications on the carboxyl group, is the process of using an alcohol (e.g., methanol or ethanol) in the presence of a catalyst, such as sodium hydroxide or potassium hydroxide, to chemically break the molecule of the raw vegetable oil into their methyl or ethyl esters with glycerol as a byproduct. Few transesterification reactions are reported with higher alcohols C8 to C14, for use as lubricant. The process of transesterification is affected by the mode of reaction, molar ratio of alcohol to oil, type of alcohol, nature and amount of catalysts, reaction animal fats by adding some functional additives[12].

Various studies have been carried out using different oils as the raw material and different alcohols (methanol, ethanol, butanol), as well as different catalysts, notably homogeneous ones such as sodium hydroxide, potassium hydroxide, sulfuric acid, and supercritical fluids or enzymes such as lipase. This thesis research work focuses on investigation of bio lubricant production using homogeneous catalyst.

1.2 Castor bean

Castor is originated in Africa and grows wild in East and North Africa, the Yemen and the Near and Middle East. It was cultivated in ancient Egypt as long as 400 B.C. Castor requires a warm climate and is killed by frost . At least 140 - 180 growing season is required before the first killing frost. It can be grown over a wide altitude range in the tropics and with both low and medium rainfall. Heavy rainfall and water logging should be avoided. At sustained temperatures above 1000 F seed may fail to set. The best soils for its cultivation are rich well-drained sandy or clayey loam's.

Up to the beginning of the 20th Century, the main use of castor was to extract castor oil to use it in the machine manufacturing, mainly as a purgative. Now the bulk of crop is utilized in industry. It is water resistant and is used for waiting fabrics and their protective coverings. It is used in the manufacture of high - grade lubricants, soap and printing inks, on textile dyeing and for preserving leather. The dehydrated oil is an excellent drying agent which compares favourable with tung oil and is used in paints and varnishes. The hydrogenated oil is used in the manufacture of waxes, plastics, carbon paper, candles and crayons. They are also used for plastics, ointments and cosmetics[22].

1.3 Statement of the problem

Ethiopia is with vary aggro ecological zone and diversity natural resources, have been known as the home land and domestication of several crop plant. Castor bean has been cultivate in large quantities in Ethiopia for several years without proper or planed way of cultivation but the country has not been using this resource. Instead of castor wasting and exporting the seeds to abroad countries, processing to useful products like renewable lubricant will minimize the foreign currency and creates job opportunity for the citizens of our society. There has been an increased worldwide environmental impact from the petroleum based lubricant derivatives usage.

1.4 Objectives

1.4.1 General Objective

Production of bio lubricant from castor oil

1.4.2 Specific objective

- ✓ Extraction of oil from castor bean.
- ✓ Characterization of physio-chemical properties(acid value, density, saponification value, free fatty acid) of extracted castor oil.
- ✓ Characterization of physio-chemical properties(viscosity, pour point, flash point, iodine value) of bio lubricant.

1.5 Scope of the project

The scope of this work is extracting oil from the castor seed by using solvent extraction and synthesize the oil to get bio-lubricant by using combined transesterification process and to characterize the extract and synthesize bio-lubricant as well as to know the yield and quality by varying parameters such as oil to alcohol ratio, reaction time and reaction temperature was analyzed.

1.6 Significance of the study

The aim of this Research is for the replacement of petroleum based lubricants, which the country is importing from different countries of the world in large amount, by biodegradable and renewable lubricant which is going to be process from non-edible plant. For the country like Ethiopia in which 85% of its people follow agriculture based way of life, such type of project is going to upgrade the society economic status.

Environmentally it has also advantage of decreasing the deposits from automobiles that use petroleum based products, wastes that are going to be release after usage which cannot be recycle back to the environment and also planting castor as farm will make comfortable environment, as it is afforestation. In this way the of petroleum reservoirs. It also brings the county to green economy system. Therefore, as stated above this paper possessed a significance area on the society, environment and country.

CHAPTER TWO

2 LITERATURE REVIEW

2.1 Bio lubricants

A lubricant is a substance introduced between two surfaces in relative motion in order to reduce the friction between them and the wear induced by contact of the surfaces. Generally, many lubricants are solutions or colloids that include a lubricating solvent or base fluid with functional additives that improve its lubrication properties[12]. Animal oils, vegetable oils, synthetic esters and mineral oils are the examples of modern lubricant base fluids. Lubricants reduce both friction and wear between moving surfaces. The measure of the ability of a lubricant to reduce friction and wear is known as lubricity.

An excellent lubricant base fluid is required to have low volatility, excellent heat and oxidative stability, good cold fluidity and high viscosity index .Bio lubricants are made from different crop oils. More than 350 oil-bearing crops are known, among which, only palm, soybean, sun flower, Jatropha, castor, coconut, sun flower, rapeseed, cottonseed, and peanut oils are considered as potential alternative bio lubricants[5].

Bio lubricants are generally considered as lubricants with high biodegradability as well as low human and environmental toxicity. The types of bio lubricant feed stock may differ from country to country and highly depend on geographical locations. In Ethiopia context, use of edible oils for bio lubricant production is unaffordable and rather illogical as Ethiopia is an importer of edible oils[5].

2.2 Historical development of lubricants

Although the use of lubricants is as old as mankind, scientific focus on lubricants and lubrication technology is relatively new. Mankind has used lubricants from the early days of civilization to assist in reducing the energy needed to slide one object against another[18]. It is recorded that grease, oil, or mud have been utilized as lubricant as early as 2400 B.C. and liquid lubricant was valuable as the original lubricant for transporting sledges in the Sumerian and Egyptian civilizations.

From the first century A.D., animal fats and vegetable oils were the principal lubricants used in machinery such as lathes, pulleys, gears. During the period of industrial revolution around 1760, heavy industrial iron and steel machinery, was

widely introduced. Animal oils such as sperm whale oil and vegetable oil from sources such as palm and groundnut oil saw increased use as lubricants. At the same time, mineral oil obtained from the distillation of coal, was developed for use as a lubricant. Subsequently, mineral oils accounted the greatest proportion of lubricant base oils. Reported that, 85–90% of the total global lubricant production originates from non-renewable petroleum precursors[8].

However, with decreasing petroleum reserves and growing environmental pollution, conventional petroleum resources are no longer preferred for many lubricant applications. A trend has developed where fractional and refined petroleum base fluid used in lubricant production is increasingly being replaced by synthetic stocks or modified plant base stock.

2.2.1 Refined petroleum base oils

Crude petroleum is a mixture that contains a large array of hydrocarbon molecules (such as paraffin, aromatics, cycloalkanes and alkenes) and small amounts of nitrogen, sulfur, oxygen, metal, and salt. Petroleum hydrocarbon molecules include covalent linked carbon atoms in an array of molecules with different carbon skeletons. Petroleum molecule carbon skeletons vary by chain length, branching patterns, cyclic structures and hydrogen saturation.

The potential utility of hydrocarbons is, in part, determined by the molecular structure. Crude petroleum oil is refined by a number of processes that reduce the average molecular weight and remove atoms such as Sulphur and nitrogen. Molecular weight reduction (cracking) and hydrogenation optimize the properties of petroleum.

2.2.2 Synthetic base oils

From 1985 to 2002, the synthetic lubricant proportion of total lubricant market for Western Europe rose gradually from less than 2% to over 10%. Synthetic fluids are categorized by their composition or their chemical structure. Typical synthetic fluids include synthetic hydrocarbons (such as polymers of olefins, chlorinated hydrocarbons, condensation products and chemically modified mineral oils), poly ether oils, esters, phosphoric acid esters, and oils containing silicon. Synthetic fluids have many advantages when compared with crude oils; however, the production cost for synthetic oil is generally four to eight times higher than mineral oil. Useful vegetable oil materials are readily available and the price of vegetable oil would be

reduced by the development of oil seeds that produce oil with enhanced stability in the future[4].

2.2.3 Vegetable oils

Historically, lubricants have been produced from both edible and non-edible vegetable oils. Most vegetable oils are predominantly TGs. TG structure provides excellent lubrication because long fatty acid chains combined with polar carbonyl groups produce high strength lubricant films. These base oils are biodegradable and, compared to mineral oils, have excellent tribological properties (low friction coefficient, good wear protection). Their range of use is limited by lower stability against thermal oxidative and hydrolytic stress and partly inferior cold flow properties. These limits can be improved gradually either with additives, or with the selection, cultivation or genetically modification of new types of plants.

Table 2.1 :Comparison of the properties of petrol based and bio based lubricant

	Petrol based lubricant	Bio based lubricant
Advantages	<ul style="list-style-type: none"> ➤ Cheap Price ➤ Abundant easily accessible satisfy global demand ➤ Longer operating time-lower downtime of machine changing lubricant. ➤ High oxidation stability 	<ul style="list-style-type: none"> ➤ Low toxicity ➤ High viscosity ➤ Higher flash point ➤ Biodegradable ➤ Less emissions Cost saving on disposal cost, storage
Disadvantages	<ul style="list-style-type: none"> ➤ Non renewable ➤ Toxic to environment Hazard disposal 	<ul style="list-style-type: none"> ➤ Poor oxidative stability ➤ Price is 1.5 - 5 times more expensive than conventional lubricants

2.2.4 Overview of Lubricant in Ethiopia

From the report on addis fortune magazine Saturday April 4, 2015, Lubtam Lubricants & Greases, the first lubricants blending plant in Ethiopia, launched production on Tuesday, June 24. Lubtam, which rests on 10,000sqm of land in Gelan town in the

Oromia Special Zone, has been established with a capital of 10 million dollars as a subsidiary of Naztech Petroleum Investment Group (NPI) based in the United Arab Emirates (UAE). The plant produces automotive, industrial and marine lubricants both for domestic consumption and export based on the standards of the American Petroleum Institute (API), according to Nazar Ibraheim (Eng.), chairman and chief executive officer (CEO) of Naztech. The company will start production with 25,000tns, 40% of which will be for domestic consumption and the rest for export to different countries in Africa, said Ibraheim during an opening speech[15].

At full capacity, the factory will produce 75,000tns – more than twice the current national demand of 35,000tns. It takes 10 barrels of crude oil to produce one barrel of lubricant, according to a presentation during the inauguration. Lubricants are imported to Ethiopia by private companies along with asphalt, with the government setting the price to which Lubtam will comply, Khidir said.

2.3 World status of the lubricant market

During the past 10 years, the global lubricant market has under gone dramatic changes. Worldwide demand for lubricants has remained at approximately 35 million tons per year since 1991. An estimated 37.4 million tons of lubricants were consumed worldwide in 2004, with 53% being automobile lubricants, 32% being industrial lubricants, 10% being process oils, and 5% being marine oils. In 2005, 37.9 million tons of lubricants were used worldwide, with the Asia Pacific region overtaking North America since 2004. However, the annual growth rate is expected to reach approximately 2% starting in 2012. The fastest growth will occur in the Asia-Pacific, with China being the major gainer[15].

2.3.1 Market opportunities for bio-based lubricants

A sustainable decision on the manufacture and use of bio lubricants must consider the economic consequences. It is normally not possible to immediately phase out the production or use of a less sustainable chemical. However, product sustainability should be a high long-term priority. The alternatives for non-sustainable substances (bio lubricants instead of mineral oils) have to be applicable in existing technology[12].

Modern manufacturing processes are often very fast (e.g. newspaper printing) or very complex (e.g. electronic devices) and production machines are fine tuned to the use of particular chemicals. Substitution of lubricants is connected to economic risks: the scarce information on proven performance of substitutes in particular applications, the practical and technical implementation of substitution, as well as the risk of lower acceptance of new products on the market. The market interest in bio-oils is characterized by the desire for immediate protection of the environment, i.e. minimizing the contamination effect in case of a severe loss of fluid. Most users do not care if the bio-oil originates from petrochemical or renewable resources. The use of bio-based automotive lubricants is so far very limited because of performance issues[21].

2.3.2 Future prospects

The fastest growth in lubricant demand in 2017 is reported in the manufacturing sector and in other markets. In fact, bio lubricants exceed the performance of mineral lubricants in terms of viscosity, low carbon-forming tendency, stability, oxidation stability, volatility requirement, and response to additives. Providing better performing lubricants for specific applications is a challenge in the lubricant industry[5].

Developing new generation heavy-duty lubricants is an example of the response of the industry to the demand for lubricated automotive equipment that will reduce environmental loading by decreasing emissions and to achieve biodegradability and non-toxicity. Bio lubricants are now widely accepted as offering a number of inherent performance advantages over conventional petroleum-based oils to formulate modern automotive engine oils[5].

2.4 Castor bean plant

Castor is originated in Africa and grows wild in East and North Africa and also other country. It can be grown over a wide altitude range in the tropics and with both low and medium rainfall. The best soils for its cultivation are rich well-drained sandy or clayey loam's. The plant itself requires relatively less fertilizers, pesticides, water and maintenance than most other cultivated crops, and it can grow in marginal land. There are different varieties of castor seeds but on the average, they contain about 46–55% oil by weight[22].

Castor seeds are poisonous to humans and animals because they contain ricin, ricin and certain allergens that are toxic. It is used in the manufacture of high - grade lubricants, soap and printing inks, on textile dyeing and for preserving leather. Castor oil is produced in the seed of the castor oil plant, *Ricinus communis*, and has been used for medicinal purposes for many years[22].

During the 20th century, a number of industrial uses were developed including its use as a lubricant. From its seeds industrialization is obtained, as main product, the oil (47%) and, as by-product, the castor waste that may be used as a fertilizer. Castor oil possess unusual and has greater density, viscosity, ethanol solubility and lubricity compared with other vegetable oil. This oil also has a wide chemical versatility inside the industry, due to be used as raw material to the synthesis of a large amount of products. Besides, castor oil presents high viscosity and low pour point, but its viscosity index is lower than the others vegetable oils, which means that its viscosity changes more with temperature than the other oils[22].

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, polyurethanes, etc. Its odd properties give lubricity to the mineral diesel, like sulfur, becoming a special oil in the current world market.

2.4.1 Properties & Chemical Composition of Castor Oil

Castor oil's chemical formula: $\text{CH}_3-(\text{CH}_2)_5-\text{CH}(\text{OH})-\text{CH}_2-\text{CH}=\text{CH}-(\text{CH}_2)_7-\text{COOH}$
Castor oil is unique among all fats and oils in that: It is the only source of an 18-carbon hydrated fatty acid with one double bond Ricinoleic acid (12-Hydroxyoleic Acid) comprises approximately 87% of the fatty acid composition Product uniformity and consistency are relatively high for a naturally occurring material. It is a toxic, biodegradable, renewable resource like other vegetable oils and animal fats, castor oil is a triglyceride, which chemically is a glycerol molecule with each of its three hydroxyl group esterified with a long chain fatty acid[21].

The chemistry of castor oil is centered on its high content of ricinoleic acid and the three points of functionality existing in the molecule. These are: The carboxyl group which can provide a wide range of esterification. The presence of hydroxyl group on castor oil adds extra stability to the oil and its derivatives by preventing the formation

of hydro peroxides. The hydroxyl groups in castor oil account for a unique Combination of physical properties: Relatively high viscosity and specific gravity, Solubility in alcohols in any proportion, Limited solubility in aliphatic petroleum solvents[21].

Table 2.2: Properties of castor oil

Property	Standards
density	0.956-0.963g/ml
Refractive index	1.477-1.479
Saponification number	1.77-1.88
Iodine value	82-88
Unsaponifiable matter	0.30-0.5%
Hydroxyl number	160mm
Viscosity@20°C	9.5-10.0dpa.s

Source:[5]

2.4.2 Castor bean oil Applications

In Ethiopia the plant has almost law use, the community use the kernel to its oily property for baking “injera”. By applying the seed kernel on hot “mitad” and make it ready for baking. Castor oil has better low temperature viscosity properties and high temperature lubrication properties than most vegetable oils, making it useful as a lubricant in jet, diesel, and race-car engines. Castor oil is the preferred lubricant for bicycle pumps, because it doesn't dissolve natural-rubber seals. Castor oil is also one of the preferred lubricants for model aircraft[9].

The lubricants company, Castrol, took its name from castor oil. For most of the lubrication purposes, the degummed variety of castor oil is the preferred grade. Used in Lubricating Grease, Aircraft Lubricants, Jet Engine Lubricants.

2.5 Methods of Seed Oil Extraction

Quality of oil depends on the type of extraction. Manufacturing of castor oil includes the Collection of raw materials for the extraction and selection of extraction method[13].

2.5.1 Batch Hydraulic Pressings

Oil seeds were processed in manual presses, where layers of grains were placed into the equipment, separated by filter cloths, filter press plates and for was applied via hydraulic cylinder. When the oil has stopped flowing, the workers opened the machine, removed the mass of crushed grain and put more fresh material[13].

2.5.2 Continuous Mechanical Pressing (Screw Press)

Among the physical processes for the extraction of castor oils, the continuous mechanical pressing emerges as the best technology to serve small farmers. That because this type of equipment associates both small scale and low cost when compared to the other methods cited. Another important advantage is the possibility of using cake resulting from the pressing as fertilizer or animal feed, since it is free of toxic solvents. The operating principle of this equipment consists a helical screw which moves the material, compressing it, and at the same time, eliminating the oil and producing the cake. Optimization of the continuous mechanical pressing consist of defining the optimum parameters, such as temperature and moisture content of grain, or adjustments on the press, in order to reach optimum yields of oil, using a minimum value of pressure applied by the press[13].

2.5.3 Super Critical CO₂ Extraction

Supercritical CO₂ extraction used for the extraction of oil seeds such as soybean, cotton seed, corn germ, rapeseed, and sunflower. The oils obtained were shown to have similar quality compared with hexane extracts; they also had lighter color and lower iron and phospholipids content resulting in a lower refining loss and reduction of subsequent refining steps[13].

2.5.4 Solvent Extraction

Extraction with solvents is the most effective method for the recovery of oil (almost 98%), rather than the previous method. When performed at low temperature, the solvent extraction has another advantage over screw-pressing: better quality of oil produced. This is because during expelling a sudden heating of the oil can occur, changing some parameters of its quality. The choice of solvent type is based on solubility of oil in the selected solvent, cost and safety[20].

2.6 Current Technologies in Bio lubricant Production

There are different technologies which are employed in the production of bio lubricant. The most common are discussed as follows.

2.6.1 Batch process system

Vegetable oil is charged to transesterification in a batch reactor in the presence of an excess amount of methanol, and catalyst[5]. An excess of methanol is necessary essentially to ensure full solubility of triglyceride and keep the viscosity of the reaction mixture low, but also for shifting the chemical equilibrium. The excess methanol is recovered for the next batch. Remaining mixture is presented to the separation of esters from glycerol which can take place either by decantation or by centrifugal. Hot water may be added to improve the phase split. The oil phase containing fatty esters is sent to finishing by neutralization with acid, followed by washing and drying. The methanol recovery takes place by flash distillation or film evaporation[5].

2.6.2 Continuous process system

Catalytic continuous process technology of fatty acid methyl Ester production is a conceptual scheme of a continuous process working at low pressure that is capable of processing a feed stock with a larger amount of free fatty acids, such as unrefined non edible vegetable oils, tallow fat and used cooking oil[5].

Table 2.3:Comparisons of batch and continuous operations

Characteristics	Batch	Continuous
Typical capital cost	Less	Greater
Economy of scale	< 10 million gallons per year	>10 million gallons per year
Feed stock flexibility	Greater flexibility	Less flexibility
Consumption of input	Greater	Less
Operating cost per gallon	Greater	Less
Product yield	Less	Greater
Typical plant size	Small	Greater

Source[12]

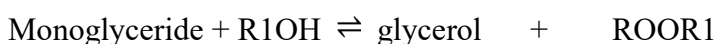
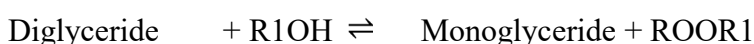
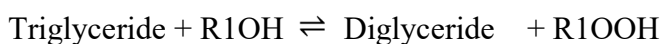
2.7 Fatty acid methyl Ester production

The main components of vegetable oils and animal fats are triglycerides, which are esters of FFA with glycerol[17]. The triglyceride typically contains several FFA, and thus different FFA can be attached to one glycerol backbone. The FFA composition is the most important factor influencing the corresponding properties of vegetable oils and animal fats. Because vegetable oils or animal fats have high viscosity.

2.8 Transesterification reaction

Transesterification is commonly used to produce FAME, in which esters are susceptible to reaction with alcohols in the presence of a catalyst so that the alcohols are exchanged.

The following reaction shows this consecutive and reversible process:



The stepwise reactions are reversible and an excess of alcohol is used to shift the equilibrium towards the formation of esters. In the presence of excess alcohol, the forward reaction is pseudo-first order and the reverse reaction is found to be second order. It's Homogeneous base and acid catalyzed transesterification and Heterogeneous acid and base catalyzed transesterification[6].

2.9 Parameters Affecting Alkali catalyzed transesterification reaction

Transesterification has been described as a chemical reaction between triglycerides and alcohol in the presence of catalyst to produce mono-esters that are termed as fatty acid methyl Ester. Transesterification reaction is affected by various parameters. The reaction is either incomplete or the yield is reduced to a significant extent if the parameters are not optimized[11].

2.9.1 Alcohol to Oil Molar Ratio

One of the most important parameters affecting the yield of the reaction is the molar ratio alcohol to triglyceride. Lower molar ratios require a longer time to complete the reaction. Excess molar ratios increase the conversion rate but leads to difficulties in the separation of the glycerol.

2.10 Catalyst

A catalyst is needed to improve the transesterification reaction and amount of yield production. The alkaline catalysts such as sodium hydroxide and potassium hydroxide are most widely used. These catalysts increase the reaction rate several times faster than acid catalysts[8].

2.10.1 Reaction time and temperature

The rate of the transesterification reaction is strongly influenced by the reaction temperature. Generally, the reaction is carried out close to the boiling point of methanol (60-65°C) at atmospheric pressure. With further increase in temperature there is more chance of loss of methanol. When the reaction temperature exceeds the boiling point of alcohol, the alcohol will vaporize and form a large number of bubbles which may inhibit the reaction[11].

2.11 Formulation of lubricant

All lubricant systems are materials formulated from one or more base fluids (native oils, synthetic esters, etc.) and property-enhancing chemical substances, commonly called additives. A base fluid means a lubricating fluid whose flow, ageing, lubricity and anti-wear (AW) properties, as well as its properties regarding contaminant suspension, have not been altered by the inclusion of additives. The quality and performance of a lubricant formulation depend on the quality of the base fluid and the combination of additives or additive package used. Combination of different additives and their quantities are determined by the lubricant type (engine oils, gear oils, hydraulic fluids, cutting fluids, way lubricants, compressor oils, etc.) and the specific operating conditions (temperature, loads, machine parts materials, environment). Industrial oils are formulated with different additives from engine oils[21].

2.12 Additives of lubricant

Additives can broadly be classified into two types that influence chemical and physical properties of base fluids (e.g. V-T characteristics, demulsibility, low-temperature properties, oxidation stability) and affect the metal surfaces (e.g. reduction of friction, increase in EP behavior, wear protection, corrosion inhibition).

2.13 Bio lubricant properties and specification standard

The main properties of a lubricant oil, which are basic requirements to the good performance of it, will be described as follows.

Viscosity: the viscosity of lubricants is the most important property of these fluids, due to it being directly related to the film formation that protects the metal surfaces from several attacks.

Pour point: The lowest temperature where is observed movement in the oil is reported as the pour point. The lower the pour point, the better the base oil, having values lower than -36°C a wide market.

Acid value (AV): this essay's goal is to measure the acidity of the lubricant, derived, in general, from the oxidation process, the fuel burning and some additives.

CHAPTER THREE

3 MATERIAL AND METHODS

3.1 Materials

3.1.1 Equipment's

Table 3.1 Equipment's and their purpose

no	Equipment	Purpose
1	Soxhlet extractor	To extract the oil from castor bean
2	Beaker	To hold,mix and heat liquid
3	Centrifuge	To separate particles suspended in a liquid
4	Reactor	Transesterification reaction in take place
5	Thermostat	To heats or cools to a set point temperature
6	Protective Equipment	To safety protection
7	Measuring cylinder	To measure the Volume required during experimentation
8	Oven	To drying castor seed
9	Separating funnel	To separating layer of immiscible liquid
10	Heater	To remove high boiling solvents under low pressure
11	Electronics balance	To measure mass and chemical will be needed
12	Burette	To how much liquid will be delivered
13	Conical flask	To hold,heat, transport and store material
14	Vibro viscometer	To measure the viscosity at a stationary or flowing state

3.1.2 Raw material and chemicals

- ✓ Castor bean
- ✓ Methanol
- ✓ potassium hydroxide

- ✓ sodium hydroxide
- ✓ Phenophtaline
- ✓ Ethanol
- ✓ Distilled water
- ✓ Water
- ✓ detergent

3.2 Method

3.2.1 Pre-treatment of castor seed

First, the raw material collected from local area and prepared for further process. Then the collected and prepared seed weighted and recorded mass is (1017gram).



Figure 3.1: Raw of castor seed

The castor seeds undergo various processing in the course of its preparation for extraction. The process that involved are: -

Clearing: The castor seed had some impurities such as sand and cover of the seed. This impurities removed by hand picking and washing[19].

Drying: Then the cleaned seeds dried in the oven at 105°C for 2hrs in order to reduce its moisture content in raw material

Winnowing: The separation of the shell from the nibs (cotyledon) carried out using tray to blow away the cover in order to achieve very high yield or decortating outer shell from castor seed by using hand to facilitate extraction of oil.



Figure 3.2: winnowing process

Grinding (size reduction): The mortar and pestle used to crush the beans into a paste (cake) in order to weaken or rupture the cell walls to release castor fat for extraction or for oil release.

3.2.2 Determination of Moisture Content of the Seed

The castor Seeds 1107g of the sample weighted and dried in an oven at 105°C for 2hrs and the weight was taken after every 2hrs[5]. The procedure repeated until a constant weight obtained. After each 2 hours, the sample removed from the oven and placed in the desiccator for 30 minutes to cool, then and re-weighed. The percentage moisture in the seed calculated by the formula:

$$\text{Moisture} = \frac{(W1 - W2)}{W2} * 100\% \quad , \text{where}$$

W1 = Original weight of the sample before drying;

W2 = Weight of the sample after drying.

3.2.3 Oil extraction from the seed by Soxhlet extraction

200ml of Ethanol poured into round bottom flask, 30g of the sample placed in the thimble and inserted in the centre of the extractor. The Soxhlet heated at 78°C and allowed for 3hrs. When the solvent is boiling, the vapour rises through the vertical tube into the condenser at the top. The liquid condensate drips into the filter paper thimble in the centre, which contains the solid sample to be extracting. The extract seeps through the pores of the thimble and fills the siphon tube, where it flows back down into the round bottom flask. This was allowed to continue for 3hrs.



Figure 3.3: Soxhlet extractor

3.2.4 Heater

After oil extracted from castor seed in Soxhlet, separation of oil from ethanol or methanol. That can be done by using heater to evaporate and adjusted at 78.5°C to separates oil from ethanol/methanol based on their vaporization level.

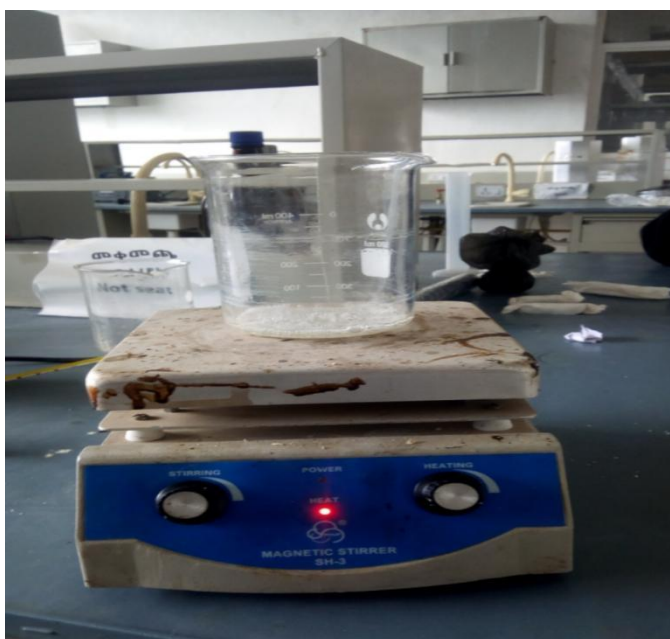


Figure 3.4: Heater

3.2.5 Purification of crude castor oil

3.2.5.1 Degumming

The extracted crude oil contains phosphatides, gums and other complex compounds which could promote hydrolysis (increase in free fatty acid) of vegetable oil during storage. During transesterification process, these compounds could also interfere. Therefore, they need to be removed by degumming process.

In this process, the crude oil heated to approximately 60°C. Hot distilled water (3%v/v of oil) added to the oil and the resulting mixture stirred well and allowed to stand for 30 minutes in separation funnel. During that process, the phosphatides present in the crude oil become hydrated and there become oil-insoluble.



Figure 3.5: Degumming

3.2.5.2 Centrifuge

The degummed oil contains some amount of water and precipitated matter. So it removed by taking the oil to the centrifuge for about 30 minute. And the hydrated phosphatides removed by centrifuge separation process.



Figure 3.6: Centrifugal

3.2.6 Physiochemical properties of castor oil

3.2.6.1 Acid Value

The 1g of NaOH required to neutralize the free fatty acid in 5 g of the sample. 5 g of the sample (castor oil) weighted and transferred into a conical flask. The weight recorded 50ml of Isopropyl alcohol and 3 drops of the (phenolphthalein) indicator solution added, then titrated with 0.5 N sodium hydroxide solution with constant stirring until a faint, pink end point appears and persist for 30 s. The volume of titrant used to reach this endpoint recorded and from the readings obtained, the acid value is evaluate using the equation below.

$$\text{Acid value} = \frac{(\text{Titrate value})(\text{Normality of NaOH})(40)}{(\text{Sample wt.})} * 100$$



Figure 3.7: Titration process

3.2.6.2 Density

An empty beaker weighted and the weight recorded, then 20ml of the sample (castor oil) poured into the beaker and weighted. From the sample weighted, the density determined by taking the ratio of the weight of the oil to the known volume 20ml in SI units according to the equation below:

$$\text{density} = \frac{\text{Sample Weight}}{\text{Sample volume}}$$

3.2.6.3 Determination of saponification value

4g of sample (castor oil) measured and placed in chemical flask. The 50ml of ethanoic sodium hydroxide which 0.1M prepared by mixing 50ml of ethanol with 0.28 gram of sodium hydroxide. Then the mixture added to the conical flask that contain sample in it. The mixture allowed boiling gently for 30min with shaking at regular interval of 2min, that boiled put in cold water for condensate. After condensate few drops (three drops) of phenolphthaline indicator added to the warm solution and then filtered with 0.5M HCl. Before starting the titrate initial volume of HCl (V_i) is 60ml. The titration placed until the pink color disappeared finally the pink color disappeared at $9v(v_f=60\text{ml})$

3.2.6.4 Percentage of free fatty acid (%FFA)

This is the percentage by weight of specified fatty acid in the oil. It is simply matter of dividing the acid value by two.

3.2.7 Bio-lubricant synthesis

Production (synthesis) of bio-lubricant involves a two stage transesterification; the first one is aimed at producing an intermediate product-methyl ester of the oils, while the Second uses the methyl ester as reactant to produce the desired product Polyester[2].

Methyl ester synthesis: the oil transesterified with methanol using potassium/sodium hydroxide as catalyst. The catalyst and glycerol part separated from the methyl Ester mixture by separator funnel after transesterification is carried out. Then, unreact methanol and trace moisture remove in oven for 5hour. The methyl ester obtained finally[2].

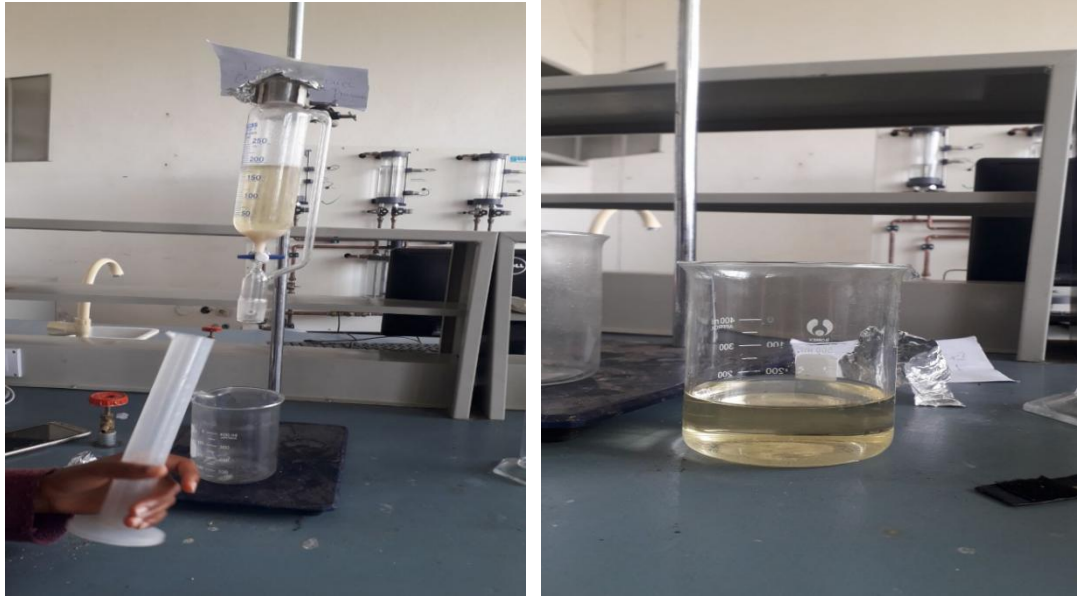


Figure 3.8: Separate funnel and its process

3.2.7.1 Experimental Design for methyl ester Production

In these work the methyl ester prepared using purified castor oil and methanol with a homogeneous catalyst of sodium hydroxide. The transesterification process variables studied were molar ratio of methanol to oil and weight percentage of catalyst, reaction period, temperature and rotation speed setted at optimum point where the maximum conversion achieved based on literature data. Atmospheric pressure used for all the runs.

3.2.9 Methyl ester Production Procedure Initially, about 20ml of castor seed oil poured into a 250ml glass reactor. The reactor assembly heated to the desired temperature by using thermostat. A measured amount of methanol and homogeneous catalyst added to the reactor. The reaction timed as soon as mechanical stirrer turned on[20].

The Transesterification carried out at temperature, optimum reaction time and rotation speed which could achieve maximum conversion for 1h. The reaction parameters chosen as follows: molar ratio of methanol to oil from 7:1, mass ratio of catalyst to oil from 1%. Finally, after transesterification carried out, catalyst and glycerol part separated from the methyl Easter mixture by separators funnel. Then, unreacted methanol and trace moisture removed in oven for 5 hour. The methyl ester obtained as a clear amber-yellow liquid[20].

Feed Material Requirement for methyl ester Production

50ml of purified castor oil used. Hence, the amount of methanol and catalyst calculated as follows using the process parameters.

Operation conditions;

Temperature = 65°C

Reaction time = 1 hr

Methanol to oil ratio = 7:1

Amount of catalyst = 1 %

Rotation speed of the stirrer = 900 rpm

3.2.10 Blending of methyl ester with additives

We have been using two additives, SLES to mix with the fatty acid methyl ester in order to improve its characteristic[6]. Before blending the ester with the appropriate additive one have to do the characterization of ester in order to know which type of additive fits to improve the characterized property. For this process 10% SLES used to mix with fatty acid methyl ester. This mixture blended until foam formation stabilizes and the additives completely solubilize in the ester by continuously supplying heat.

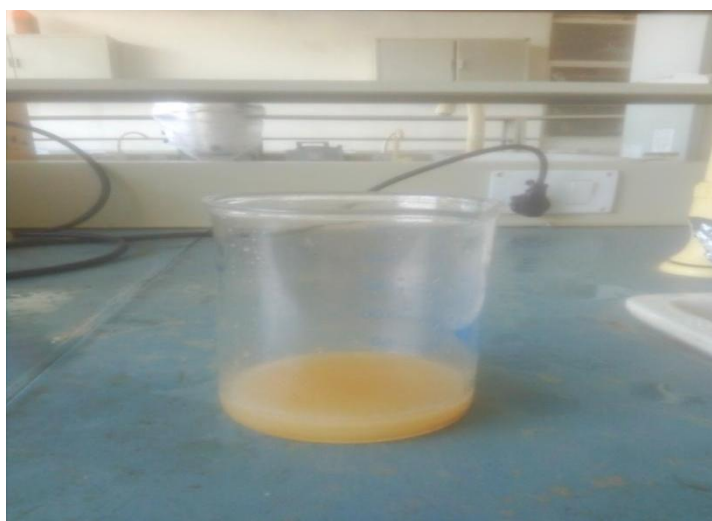


Figure 3.9: Bio lubricant

3.2.11 Characterization of bio-lubricants

3.2.11.1 Viscosity

Viscosity is more commonly known as resistance to flow. If lubricating oil is considered as a series of fluid layers superimposed on each other, the viscosity of the oil is a measure of the resistance to flow between the individual layers. A high viscosity implies a high resistance to flow while a low viscosity indicates a low resistance to flow. Viscosity varies inversely with temperature and directly with pressure[12].

3.2.11.2 Pour Point

The Pour Point is the lowest temperature at which oil will flow. This property is crucial for oils that must flow at low temperatures. A commonly used rule of thumb when selecting oils is to ensure that the Pour Point is at least 10°C lower than the lowest anticipated ambient temperature[12].

3.2.11.3 Iodine Value

Iodine value is the measure of the degree of unsaturation in relation to the amount of fat or oil. Iodine value is defined as the gram of iodine absorbed per 100g sample. When unsaturated oil heated, polymerization of the triglyceride occurs which leads to gum formation. Also, unsaturated compounds are susceptible to oxidation when expose to air, there by degrading the oil quality. Hence, the higher the iodine value, the greater the degree of unsaturation[12].

3.2.11.4 Flash Point

The Flash Point is the lowest temperature, to which a lubricant must be heat before it vaporized, when mix with air, ignited but not continue to burn. The Fire Point is the temperature at which lubricant combustion sustained. The flash and fire points are useful in determining a lubricants volatility and fire resistance[12].

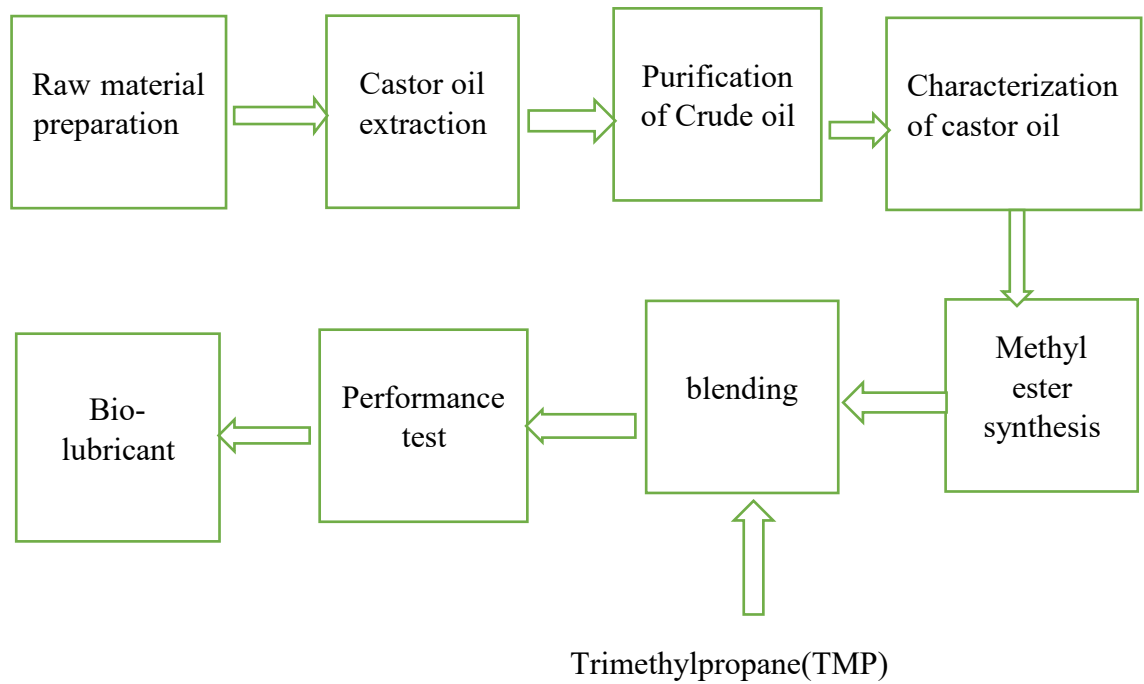


Figure 3.10:Flow diagram of bio-lubricant production

CHAPTER FOUR

4 RESULTS AND DISCUSSION

4.1 Seed preparation

For 1017g of castor seeds 255g husk carefully removed. After removing the husks and the wastes, 762g of castor seed obtained.

$$\text{Moisture content} = \frac{(\text{Mass of castor seed before dry} - \text{Mass of castor seed after dry})}{\text{Mass of castor seed before dry}} * 100\%$$

$$\text{Moisture content} = \frac{1017g - 1005g}{1017g} * 100\%$$

$$\text{Moisture content} = 1.1799\%$$

Generally the amount of heat leave from sample are increase as duration of time in oven increased. Also moisture content of sample affected by duration of time and level of temperature to be set.

4.2 Castor oil Extraction

For extraction of oil from castor bean by using two solvent which are ethanol or methanol. The product of the same sample and the same mass was different in yield amount for different solvent.

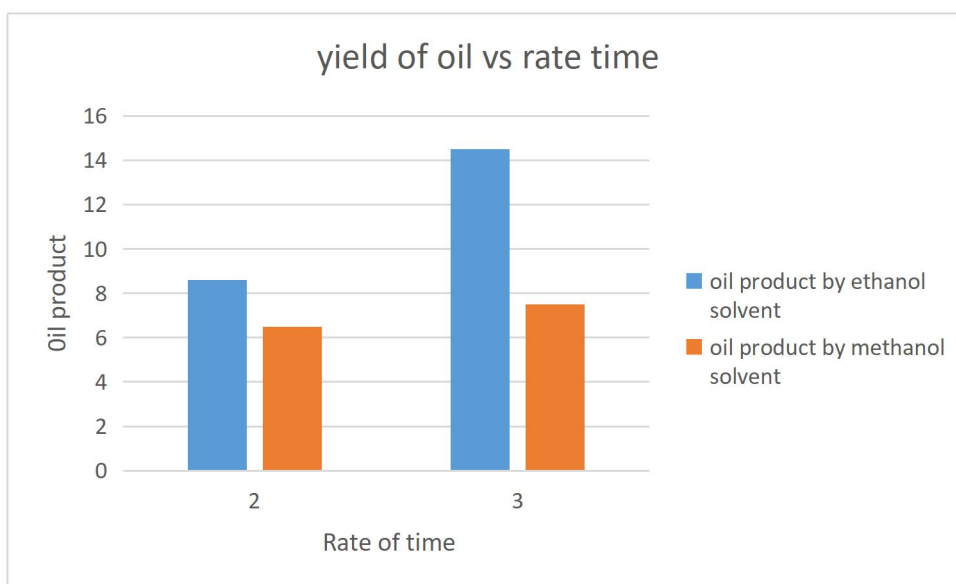
Experiment

Table 4.1: extraction of castor oil from using ethanol or methanol as a solvent.

Exp no	Mass of sample	Volume of solvent	Extraction time	Set temperature	Oil product	Byproduct mass
Exp1	20g	134ml(ethanol)	3hr	78°C	14.5g	9.34g
Exp2	20g	134ml(ethanol)	3hr	70°C	10.2g	11.5g
Exp3	20g	134ml(ethanol)	2hr	78°C	8.6g	13.7g
Exp4	20g	134ml(methanol)	3hr	65°C	7.5g	14.4g
Exp5	20g	134ml(methanol)	3hr	60°C	6.7g	15.3
Exp6	20g	134ml(methanol)	2hr	65°C	6.5g	14.9g
Exp 7	20g	134ml(methanol)	3hr	5°C	0	20g

4.3 Effect of Extraction Time on the Yield of Castor Oil

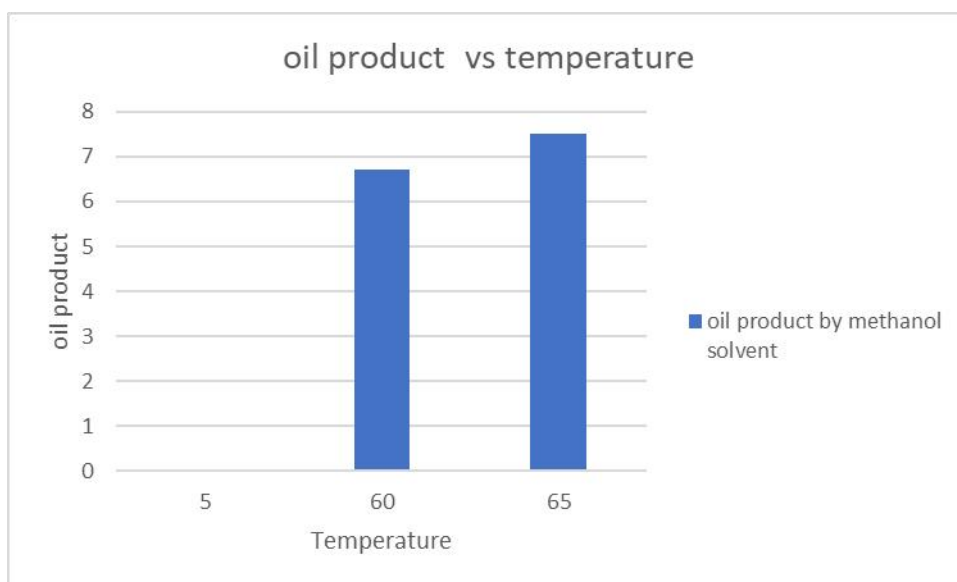
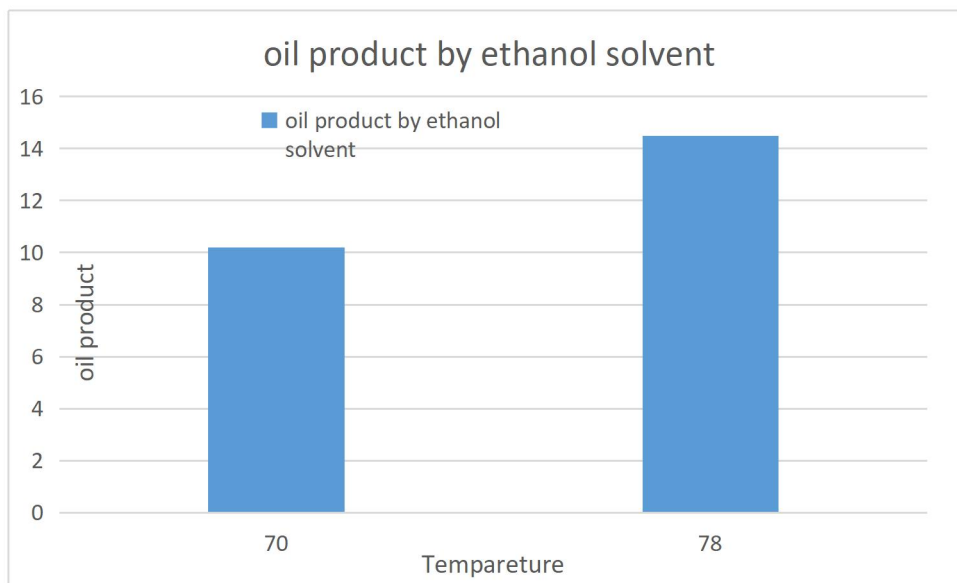
From the parameters extraction time also affects the yield of oil in proportional ways that means the yield of oil increased as extraction time is increased. Extraction time plays a great role on the percentage yield of castor seed oil using ethanol or methanol as a solvent. The above table shows that as the contact time increases the oil yield also increases this continues till transfer of oil from the castor bean powder to the solvent attains zero. In other word, when the maximum amount of extract oil is obtained, the oil yield level remains invariable even by extending the reaction time. So that in the Soxhlet extraction the maximum oil yield could be finding at an extraction time of 3hours and above.



Graph of oil product vs time variation

4.4 Effect of temperature on yield of oil

Temperature is one of the most parameter that affects the yield of oil extracted from castor seeds. From the above table for small value of temperature and also small amount of oil was obtained. The yield of castor oil is the maximum at 78°C by ethanol solvent and 65 °C by methanol solvent. This indicates that after the maximum temperature obtained; increase the temperature above it maximum affect yield of oil by formation offside product.



Graph of oil product vs temperature variation

4.5 Determination of physical properties of castor oil

4.5.1 Determination of PH value

PH of castor oil is measured by PH electrode after the pH electrode standardized by buffer solution, then pH value of oil is 6.125.

4.5.2 Acid Value

The 5g of NaOH required to neutralize the free fatty acid in of the sample. 5 g of the sample (castor oil) weighted and transferred into a conical flask. The weight recorded 50ml of isopropyl alcohol and 3 drops of the (phenolphthalein) indicator solution added. Then titrated with 0.5 N sodium hydroxide solution with constant stirring until a faint, pink end point appears and persist for 30 s. The volume of titrant used to reach

this endpoint is recorded and from the readings obtained, the acid value is evaluate using the equation below.

$$\text{Acid value} = \frac{(\text{Titrate value})(\text{Normality of NaOH})(40)}{(\text{Sample wt.})} * 100$$

$$AV = \frac{(41\text{ml} - 24\text{ml})(0.1) * 40\text{g/mol}}{5\text{g}} * 100 = \frac{1360}{1000} = 1.36$$

4.5.3 Percentage of free fatty acid (%FFA)

Before Acid value calculated, 0.1N of sodium hydroxide prepared from 1g of powder of NaOH and 250ml of distilled water. Then 10g of sample, 25ml of ethanol and 4-5 drops of phenolphthalein mixed in beaker and titrated by using NaOH up to the pink color is appeared. Then free fatty acid value calculated;

$$\text{FFA} = M_w * \frac{(C * V)}{m} = 40 * \frac{0.1 * 0.25}{10\text{g}} = 0.1$$

4.5.4 Determination of saponification value

4g of sample (castor oil) measured and placed in chemical flask. The of ethanoic sodium hydroxide 0.1M prepared by mixing 50ml of ethanol with 0.28 gram of sodium hydroxide. Then the mixed one added to the conical flask that contain sample in it. The mixture allowed boiling gently for 30min with shaking at regular interval of 2min. that was boiled put in cold water for condensate. After condensate few drops (three drops) of phenolphthaline indicator added to the warm solution and then filtered with 0.5M HCl. Before starting the titration initial volume of HCl (Vo) is 40ml. The titration take placed until the pink color disappeared. Finally the pink color disappeared at 60ml. Then saponification value (Sv) calculated by:

$$SV = 1000 * 0.028 * \frac{(V_f - V_i)}{m} = 1000 * 0.028 * \frac{(60\text{ml} - 40\text{ml})}{4\text{g}} = 140$$

4.5.5 Density

An empty beaker weighted and the weight is recorded, then 20ml of the sample (castor oil) poured into the beaker and weighted. From the sample weighted, the density determined by taking the ratio of the weight of the oil to the known volume (20ml) in SI units according to the equation below.

$$\text{Density} = \frac{\text{mass of sample}}{\text{volume of sample}} = \frac{18\text{g}}{20\text{ml}} = \underline{0.9\text{g/ml}}$$

Table 4.2: Experimental result of physical property of the extracted castor oil.

Property	Experimental value	ASTM specification of Quality Castor Oil
Density(g/ml)	0.9	0.945-0.965
Saponification(mg KOH/g of oil)	140	175-187
Free fatty acid	0.1	≤ 0.2
Acid Value (mg KOH/g)	1.36	0.4-4.0

These properties make the extracted castor oil a reliable feedstock for bio-lubricant synthesis. The composition of Free Fatty Acid of the oil was below the 2%. From this result the base catalyzed transesterification reaction was performed without any side reaction, soap formation, as a result the separation of glycerol from the fatty acid methyl Ester was simple which was going to minimize extra cost of production of bio lubricant[4].

4.6 Determination of physio-chemical properties of bio-lubricant

The viscosity of the bio-lubricant at 40 °C and 80°C are very important lubricity properties; they are useful in determining the fluidity of the lubricant at low and high temperatures. They also show the thermal stability of the lubricant. This may be due to addition of oxygen molecule in the midst of double bond, thereby increases the inter-molecular forces, molecular weight and polarity. But viscosity of the synthesized castor oil bio-lubricant found to be slightly lower than the requirement of the ISO VG 46. Acid value (AV) of COFAME epoxide founded to be 0.49 mg NAOH/g. Lower acid value of epoxide sample assures that trouble free operation of the equipment on its utilization and result low corrosion. During that process density of epoxidized COFAME (956 kg/m³) founded to be higher than its unmodified COFAME (930 kg/m³).

Similarly, kinematic viscosity (at 40 °C) of COFAME epoxide was two and half times higher than COFAME. This may be due to addition of oxygen molecule in the midst of double bond, thereby increases the intermolecular forces, molecular weight and polarity. The iodine value analyzed to indicate the remaining unsaturation of COFAME after the epoxidation reaction in the product. The reductions of iodine values indicated the consumption of unsaturation during epoxidation.

The flash point is useful in determining the volatility and fire resistance of a bio-lubricant. It is used to determine the transportation and storage temperature requirements for bio-lubricants. In that process, the lowest flash point (155°C) is achieved. The unsaturation content has a positive influence on the flash point, i.e. higher the unsaturation content lowers the flash point and vice versa.

Table 4.3: Properties of synthesized bio-lubricant standards according to ISO VG-46

Property	Bio-lubricant	ISO VG-46
Density(g/ml)	0.8	0.92
Viscosity @80°C	424	>450
Acid value	0.49	<1
Iodine value	9.11	<8
Flash Point	170	>170

4.7 Effect of Reaction Temperature

Temperature has significant effect on the yield of oil. At temperature of process 78°C for ethanol solvent(134ml), process time 3hr, 20g of castor seed and the oil achieved is 14.5g. Also at 70°C for ethanol solvent(134ml), process time 3hr, 20g of castor seed and the oil achieved is 10.2g. For methanol solvent(134ml), temperature 65 °C , process time is 3hr, 20g of castor seed and the oil achieved is 7.5g. For the same solvent methanol(134ml), temperature 60°C, process time is 3hr, 20g of castor seed and the oil achieved is 6.7g. The increasing or decreasing of temperature from its maximum, affect the yield of oil for both solvent(ethanol and methanol) at constant extraction time.

4.8 Effect of extraction time

Time is an important parameter that affects the extraction of oil. For 20g of castor seed, ethanol(134ml), process time 3hr and the achieved oil is 14.5g at 78°C. And also for 20g of castor seed, ethanol solvent(134ml), process time 2hr and achieved oil 8.6g at 78 °C . For methanol solvent(134ml), 20g of castor seed, process time 3hr and achieved oil 7.5g at 65°C. for the same solvent methanol(134ml), 20g of castor seed , process time 2hr and oil achieved 6.5g at 65°C. Generally time variation affect the yield of oil for both solvent(ethanol and methanol) at constant temperature.

CHAPTER FIVE

5 MASS AND ENERGY BALANCE

Material and energy balance are very important in the chemical process industry. Material and energy balance states that mass and energy can neither be created nor destroyed. Statements based on the law of conservation of mass such as “total mass of input is equal to the total mass of output”. The design of a new process or analysis of an existing one is not complete until it is established that the inputs and outputs of the entire process and of each individual unit satisfy balance equations.

5.1 Mass Balance

general material balance

$$\text{Input} + \text{Generation} - \text{Output} - \text{Consumption} = \text{Accumulation}$$

Assumptions

- For steady state systems accumulation is zero.
- For non-reactive species generation and consumption is zero.

Batch process

- Plant uses 1000kg/hr

Working day is 300days per year.

1. The moisture content of castor seed calculated by using the following formula %

$$\% \text{ Moisture content} = \frac{(W_1 - W_2)}{W_2} * 100$$

Where:

W1 – the weight of castor seed before drying

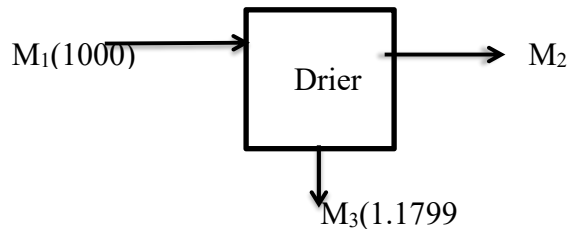
W2 – the weight of castor seed after drying

$$\% \text{ Moisture content} = \frac{1017g - 1005g}{1017g} * 100\%$$

$$\% \text{ moisture content} = \underline{1.1799\%}$$

5.1.1 Mass Balance on Drier

From general material balance on the drier:



From general material balance:

Input = out put

$$M_1 = M_2 + M_3$$

$$M_3 = 1.1799\% * M_1 = 0.11799 * 1000 \text{ kg/hr}$$

$$M_3 = \underline{11.76 \text{ kg/hr}}$$

$$M_2 = 1000 \text{ kg/hr} - 11.76 \text{ kg/hr} = \underline{988.21 \text{ kg/hr (sample dried)}}$$

The weight of the outer shell removed after separated from the castor seed was calculated by:

$$\% \text{ cover seed} = \frac{w_i - w_f}{w_i} * 100\%$$

Where, w_i = weight before remove seed shell.

w_f = weight after shell was removed.

$$\% \text{ cover seed} = \frac{(1005 - 760)}{1005} * 100\% \\ = \underline{24.378\%}$$

5.1.2 Extractor (soxhlet extraction)

In laboratory for 30g of castor seed was grinded is required for extract oil by solvent. Thus 200ml of solvent was used. In this form 1000kg/hr. of castor seeds 6666L of solvent will be required. Then:

Mass of solvent = density of solvent * volume of solvent

Density of ethanol and methanol at it's boiling point.

Density of ethanol at 78°C is 0.66 g /ml

$$0.66 \text{ g/ml} = 0.66 \text{ kg/l}$$

$$\text{Mass of ethanol} = 0.66 \text{ kg/ml} * 200 \text{ ml} = \underline{13200 \text{ kg/hr}}$$

Density of methanol at 65°C is 0.275g/hr

$$0.275 \text{ g/ml} = 0.275 \text{ kg/l}$$

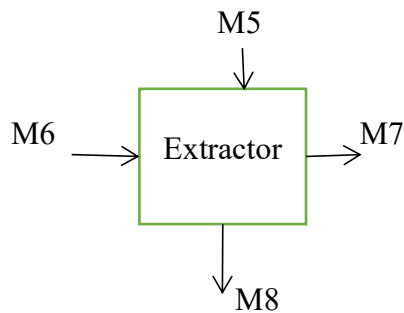
$$\text{Mass of methanol} = 0.275 \text{ kg/ml} * 200 \text{ ml}$$

$$= \underline{0.055 \text{ kg/hr}}$$

Then the amount of solvent used for the grinded was:

$$\text{Mass of ethanol} = 0.66 \text{ kg/l} * 6666 \text{ l} = 4399.56 \text{ kg/hr}$$

Mass of grinded castor bean=760kg/hr



Where, M5=amount of solvent

M6=sample of castor bean

M7=extracted oil and solvent

M8=byproduct

Where, M5=amount of solvent

M6=sample of castor bean

M7=extracted oil and solvent

M8=byproduct

Total mass of inlet=mass of ethanol + mass of castor bean

$$4399.56\text{kg/hr} + 760\text{kg/hr} = \underline{5159.56\text{kg/hr}}$$

Over all mass balance

$$M4 + M5 = M6 + M7$$

Balance for castor bean sample(M6)

$$M5 \cdot x_{m5} + M6 \cdot x_{m6}$$

We feed concentration $x_{m5} = 0.506$ and that not solid particle included in the over flow so,

$$X_{m7} = \text{zero flow } m5 \cdot x5 + m7 \cdot x7 = 5159.56\text{kg/hr} \cdot x_{mt}$$

$$760\text{ kg/hr} \cdot 0.506 + 0 = 5159.56\text{kg/hr} \cdot x_{mt}$$

$$x_{mt} = 0.0745 = x_A, \text{ where } x_A \text{ is mass fraction of solid In total mass inlet.}$$

Balance for solvent (M5)

We feed concentration $x_{m6} = 0.4934$, Pure ethanol is used as solvent $x_{m6} = 0$ (have no oil in it)

$$M_5 \cdot x_5 + M_6 \cdot x_6 = M_t \cdot x_{mt}$$

$$760 \text{ kg/hr} \cdot 0 + 4399.56 \text{ kg/hr} \cdot 0.4934 = 5159.56 \text{ kg/hr} \cdot x_{mt}$$

$$x_{mt} = 0.42 = x_B \text{ where, } x_B \text{ is mass fraction}$$

of oil Mass fraction solvent in mixing $x_A +$

$$x_B + x_{sol} = 1$$

$$x_{sol} = 1 - 0.42 = 0.58$$

$x_{sol} = 0.5055$ (mass of fraction in total mass) the

amount leaving mass M_t

$$x_A = M_7 \cdot x_{m7} + M_8 \cdot x_{m8}$$

from $x_{m8} = 0$ (no solid

particle) $x_{m6} = 1 - 0.5055$

$$x_{m6} = 0.4945 \text{ from total}$$

$$\text{mass balance } M_t = M_7 + M_8$$

$$\text{but, } M_7 = 5159.56 \text{ kg/hr} \cdot$$

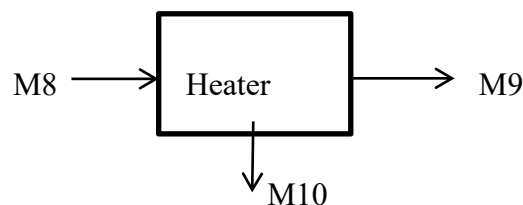
$$0.506 / 0.4945$$

$$M_7 = 5276.54 \text{ kg/hr.}$$

$$M_8 = M_t - M_7 = 5159.56 \text{ kg/hr} - 5276.54 \text{ kg/hr.} = 119.98 \text{ kg/hr.}$$

the amount of solvent and oil whose 5276.54 kg/hr. and separate by using rotary evaporator and the solvent.

5.1.3 Mass Balance on Heater



Where, M_8 = mass of oil and solvent mixture

M_9 = mass of solvent

M_{10} = mass of oil

$$\% \text{ Oil yield} = \frac{(\text{mass of oil and solvent} - \text{mass of solvent})}{\text{mass of oil and solvent}} * 100\%$$

$$\% \text{ of oil yield} = \frac{(150 - 130)}{150} * 100\% = \underline{13.33\%}$$

From the result we obtained from laboratory the solvent removed from rotary evaporator is 86,67% and oil extracted is 13.33%.

$$M8 = M9 + M10 ; \text{ but } M9 = 0.8667 * 119.98 \text{ kg/hr.}$$

$$M9 = \underline{103.98 \text{ kg/hr}} \text{ (mass solvent)}$$

$$\text{where, } M10 = M8 - M9$$

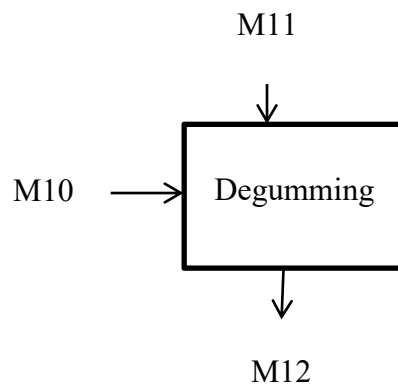
$$M10 = 119.98 \text{ kg/hr} - 103.98 \text{ kg/hr} = \underline{16 \text{ kg/hr}} \text{ (mass of oil)}$$

5.1.4 Mass Balance on degumming

The amount of water added for degumming is 3% of the Cleaned oil to remove phosphatides, gums and other complex compounds that contains in castor oil.

$$\text{Amount of water required for degumming} = 0.03 * M10$$

$$= 0.03 * 16 \text{ kg/hr} = \underline{0.48 \text{ kg/hr}}$$



Where, M11=water, M10=oil, M12=Cleaned oil+water.

$$M10 = 16 \text{ kg/hr}, M11 = 0.48 \text{ kg/hr}$$

$$M12 = M10 + M11 = 16 \text{ kg/hr} + 0.48 \text{ kg/hr} = \underline{16.48 \text{ kg/hr}}$$

5.1.5 Mass balance on Centrifuge

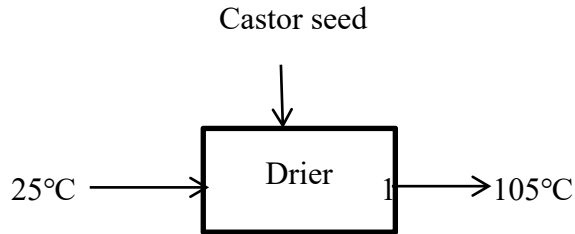
From the degumming oil 10% (w/w) of gum and water is removed by centrifuge as it has been checked in laboratory works.

$$\text{Amount of gum and water removed} = 0.1 * 16.48 \text{ kg/hr.} = \underline{1.648 \text{ kg/hr.}}$$

Purified oil = 16.48kg/hr- 1.648kg/hr. = 14.832kg/hr.

5.2 Energy Balance

5.2.1 Energy Balance on Drier



$Q = Mcp\Delta T$, where Q is energy required (KJ)

ΔT is temperature difference (k)

M is mass (kg)

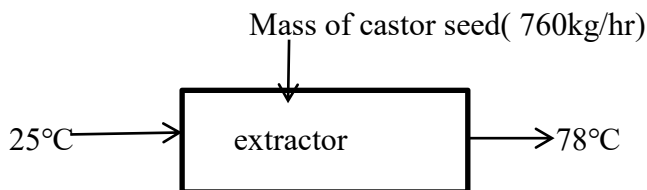
C_p is specific capacity
(kj/kg*k)

Mass of castor seed =
1000kg/hr

$T_{in} = 25^\circ C; T_{out} = 105^\circ C$

$Q = 1000\text{kg/hr} * 2.61\text{kJ/kg*k}(105-25) = \underline{208800\text{KJ/hr}}$

5.2.2 Energy Balance on Soxhlet Extractors



$Q = Mcp\Delta T$

Mass of castor bean = 760kg/hr

Mass of Ethanol = 4399.56kg/hr

C_p of ethanol = 2.575KJ/kg.k

C_p of castor bean = 2.61KJ/Kg.K

$Q_T = Q_1 + Q_2$, where Q_1 = heat energy needed for castor bean

Q_2 = heat energy needed for ethanol

$Q_1 = M_1 cp\Delta T = 760\text{kg/hr} * 2.61 * (78-25) = \underline{105130.8\text{KJ/hr}}$

$Q_2 = M_2 cp\Delta T = 4399.56 * 2.55 * (78-25) = \underline{594600.534\text{KJ/hr}}$

$$Q_T = Q_1 + Q_2 = 105130.8 \text{ KJ/hr} + 594600.534 \text{ KJ/hr} = \underline{699731.334 \text{ KJ/hr}}$$

5.2.3 Energy balance on Heater



At laboratory level, from 20g of castor bean sample and 134ml of ethanol solvent, we achieved 16.5g of castor oil

At level of plant capacity, the raw castor bean of 1000kg/hr

Oil yield = 16kg/hr

$$C_P = C_{P \text{ oil}} \cdot X_{\text{oil}} + C_{P \text{ ethanol}} \cdot X_{\text{ethanol}}$$

$$X_{\text{oil}} = \frac{M_{\text{oil}}}{M_{\text{total}}} = \frac{16}{120} = \underline{0.1333}$$

$$X_{\text{ethanol}} = 1 - 0.1333 = \underline{0.867}$$

$$C_P = 0.133 \cdot 1.82 \text{ KJ/kg.k} + 0.867 \cdot 2.575 \text{ KJ/kg.k} = \underline{2.474 \text{ KJ/kg.k}}$$

$$Q = M C_P \Delta T = 120 \text{ kg/hr} \cdot 2.474 (78 - 25) = \underline{15734.64 \text{ KJ/hr}}$$

CHAPTER SIX

6 CONCLUSIONS AND RECOMMENDATION

6.1 Conclusions

The characterization of the extracted oil like density 0.9 g/ml, moisture content 1.1799%, Acid Value (NaOH/g oil) 1.36, Composition of Free Fatty Acid (%wt.) 0.1 and oil yield 13.33% was obtained. Then transesterification reaction was done at a temperature of 65-67°C, 1.0% (w/w) KOH catalyst amount and for 7:1 molar ratio of alcohol to oil. The yield of this reaction was 98%. Also the extraction or oil yield is affected by increasing and decreasing of temperature, duration of time for extraction process. Generally oil yield achieved is maximum at optimum temperature and time. After blending the FAME with additives, SLES and the physicochemical properties of the bio lubricant, Acid Value 0.49 , viscosity 424mm²/s, and flash point 170 °C obtained. Therefore, castor can be used as a less expensive supplementary feedstock for bio lubricant production by curing the environment and increasing the agricultural earning.

6.2 Recommendation

The oil extracted using soxhlet extraction; though, this type of extraction method is time consumed and has very low efficiency and yield. For this reason, soxhlet with better efficiency and design is recommended since it has lower cost than others like mechanical press and supercritical extraction. Bio lubricant produced from non-edible castor oil which are available locally. Consequently, consideration must be given to produce bio lubricant as substitute of petroleum based lubricant. And also castor bean cake not considered as useful product. It is can be used as fertilizer, fungicide, in plant parasitic nematode control, in animal feed (if detoxified), in recovery of depleted soils and raw material for ethanol production. So it should be considered for further processing. It also recommended that the country should do further agricultural research about castor bean plant, import castor bean that has a good yield of castor oil, and reduce the tax loaded for importing equipment and chemicals needed for the investment in order to encourage investors who are interested to work on bio lubricant production.

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