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Production of Biolubricant from Castor (Ricinus) Oil

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***Abstract* – The increasing prices of crude oil, the depletion of crude oil reserves in the world, and global concern in protecting the environment from pollution drive for searching lubricants from alternative bio sources. A bio lubricant is a renewable lubricant that is biodegradable, non-toxic and has net zero greenhouse gases. The objective of this study is to use non edible oil like castor oil as bio lubricant in order to utilize renewable resource and to solve environmental issues related to petroleum lubricants. In this study, a base catalyzed method was successfully used in the synthesis of base oil (FAME) from castor seed oil. The study uses KOH catalyzed transesterification in which other variables affecting the acid value and the methyl ester yield, such as molar ratio, catalyst concentration, reaction time and reaction temperature, were analyzed according to studies of different literature review to determine the optimum yield of FAME from the seed oil. The important properties of the base oil (density, kinematic viscosity, acid value or FFA composition, moisture content,) were compared to those of ASTM and EN standards for the FAME. The comparison shows that the castor seed oil methyl Easter could be used as an alternative base oil for bio lubricant.**

***Keyword* – Bio Lubricant, Biodegradable, FAME, Non-Edible Oil, Transesterification,**

I. INTRODUCTION

1.1 Background

Formulations made from vegetable based oils together with corresponding additives are usually coined "bio lubricants" which has final composition of 60–99% of base oil and the remaining as additive, depending on the desired performance [1, 2].

Recently, increasing attention to environmental issues has driven the lubricant industry toward ecofriendly products from renewable sources. The use of biodegradable and environmentally accepted lubricants from vegetable oil has increased over the past 25 years [3]. However, millions of tons of lubricants are dumped into the environment through leakage and careless disposal. Some of these wastes are resistant to biodegradation and are threats to the environment. Thus, there are two major issues confronting the lubricant industries: the search for raw materials that are renewable and products that are biodegradable [4].

Vegetable oils are mainly triglycerides which contain three hydroxyl groups and long chain unsaturated free fatty acids attached at the hydroxyl group by ester linkages [5, 6]. 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 [7].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 [8]. Chemical modifications may improve the thermal, oxidative and hydrolytic stabilities of the vegetable oils. The most important modifications occur on the carboxyl groups of the fatty acids, approximately 90%, while oleochemical reactions on the fatty acid chain are approximately 10% [5].

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 lubricants. [9].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 time, and temperature [10].

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 lipases. This thesis research work focuses on investigation of bio lubricant production using homogeneous catalyst.

1.1.1 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 [11]. It is recorded that grease, oil, or mud have been utilized as lubricant as early as 2400 B.C. and liquid lubricant wasvaluable as the original lubricant for transporting sledges in the Sumerian and Egyptian civilizations [12].

1.1.2 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 [13]. In 2005, 37.9 million tons of lubricants were used worldwide, with the Asia-Pacific region overtaking North America since 2004. A 2007 Fredonia report stated that 41.8 million tons of lubricants were consumed in 2007, and the overall growth rate was 0.8%. Limited data are available in the market for 2008. 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.

1.1.3 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 factory has two hangars, which were built by DerejeYemaneberhan Construction, a grade six contractor, under the supervision of German Mirror, a UAE company which also designed the factory.

1.1.4 Future prospects

The increase in world lubricant demand will be aided by the current expansion of the rebound in manufacturing and other industrial activities because of the ongoing rapid industrialization and the increasing vehicle ownership rates, particularly in China. These trends will also favor growth in Africa, the Middle East, and Latin America. 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 [14]. 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 [15]. 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.

1.2. Statement of the problem

Ethiopia, endowed with varied agro ecological zone and diversified natural resources, have been known as the home land and domestication of several crop plant. Castor bean has been cultivated in large quantities in Ethiopia for several years without proper or planed way of cultivation but the country has not been using this resource.

According to international energy report, February 2015, Ethiopia is exporting the castor seed to abroad countries and importing back the different products of it. For instance an average of 505,734.00 barrels per day of petroleum products is imported from different countries since 1986 to 2010. Those reasons were main point of interest for this project is instead of wasting and exporting the seeds to abroad countries, processing into useful products like renewable lubricant will minimize the foreign currency and creates job opportunity for the citizens of our society. In the last decades, there has been an increased worldwide concern about the environmental impact from the petroleum derivatives usage. Although only approximately 1% of all consumed petroleum be used on the lubricants formulations, the most part of these products are disposed in the environment without any treatment. For instance, 1 liter of mineral oil contaminates 1 million liters of water for the human consumption and this issue has driven the biodegradable lubricants development [16].

1.3 General Objective

The general objective of this paper is production of bio lubricant from castor bean.

1.3.1 Specific objective

- To Measure Viscosity, density and acid number of the castor oil
- To trans esterify the castor oil
- To blend the ester with SLES and cocamidopropylbetaine additives.
- To evaluate viscosity, pour point and density of bio lubricant

1.4. Significance of the study

This paper is aimed at the replacement of petroleum based lubricants, which the country was importing from different countries of the world in large amount, by biodegradable and renewable lubricant which is going to be processed from non-edible plant i.e. castor.

By doing so the first beneficial will be farmers who are going to prepare a farm of this plant. This is because they will be raw material suppliers for the industries that are engaged in the production of castor based lubricants. 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 released after usage which cannot be recycled back to the environment and also planting castor as farm will make comfortable environment, as it is afforestation.

This type of project will initiate the country to minimize importing petroleum based lubricants by replacing them with biodegradable and renewable lubricant which has better characteristic than the recent one which will in turn decrease cost of importing, transportation. In this way the country will become energetically self-sufficient, no fear for the depletion of petroleum reservoirs. It also brings the county to green economy system.

Therefore, as stated above this paper will possess a significance area on the society, environment and country.

II. MATERIAL AND METHOD

2.1 Materials and Equipment

The raw materials used during the experiment were castor oil, methanol, potassium hydroxide as a homogeneous catalyst and test study chemicals was also sodium hydroxide, methanol alcohol, phenophtaline, distilled water for acid value test, water and sodium hydroxide for oil degumming and for neutralization respectively. The raw materials was obtained from the laboratory of school of Chemical and bio Engineering, Addis Ababa institute of technology.

The equipment used during the experimentations were mechanical oil extractor machine, beaker, centrifuge, Glass reactor equipped with mechanical stirrer, thermostat, condenser, separating funnel, Rotary Evaporator, balance,

burette, water bath, conical flask, Vibro viscometer, test tube, vacuum pump.

2.2 Experimental method

2.2.1 Castor seed preparation

The castor seed had some impurities such as sand and cover of the seed. This impurities were removed by hand picking and was washed by tap water. Then the prepared seeds were dried in the oven at 105^0c for 2hrs in order to reduce its moisture content.

2.2.2 Oil extraction

The oil was extracted using mechanical pressing, castor seed was fed in to mechanical presser which exerts high pressure for extracting the oil. When oil was exerted, solid cake was remain in the unit which was removed after one cycle of extraction by dismantling the equipment; oil was collected in a different side. Since there was some amount of impurities along with the oil, the extracted oil was further filter by passing through sieve to filter.

2.2.3 Purification of crude castor oil

2.2.3.1 Degumming

The oil was heated to 70^0C under stirring at 1000 rpm in a jacketed glass vessel connected to a circulation thermostat, and using 3% of boiled distilled water. The mixture was stirred for 60 minutes and removed.

2.2.3.2 Centrifuge

The degummed oil contains some amount of water and precipitated matter. So it was removed by taking the oil to the centrifuge that was operated for about 30 minute.

2.2.4 Characterization of Purified castor Oil

2.2.4.1 Acid value

Five gram of the sample (castor oil) was weighed and transferred into a conical flask. The weight was recorded.50ml of isopropyl alcohol and 3 drops of the (phenolphthalein) indicator solution were added. It was then titrated with 0.5 N potassium hydroxide solution with constant stirring until a faint, pink end point appears and persisted for 30 s. The volume of titrant used to reach this endpoint was recorded and from the readings obtained, the acid value is evaluated using the equation below.

$$Acid\ Value = \frac{(Titre\ value)\ (Normality\ of\ NaOHt)(40)}{(Sample\ wt.)} * 100$$

2.2.4.2 Percentage free fatty acid (%FFA)

The percentage free fatty acid (%) was calculated dividing the acid value by two.

2.2.4.3 Density

The density of the oil was measured by measuring the empty beaker weight and the weight was recorded, then 50 $cm^3$of the sample (castor oil) was poured into the beaker and weighted. From the sample weighted, the density was determined by taking the ratio of the weight of the oil to the known volume (50 cm^3) in SI units according to the equation below:

$$Density = \frac{Sample\ weight}{Sample\ volume}$$

2.2.4.4 Viscosity

The viscosity of the oil sample was determined at temperatures of 40 ^{0}C and 22.5 °C using vibro viscometer. Initially the sample was at 22.5 ^{0}C and the viscosity was measured directly by taking it in to the instrument. Then the oil was heated to reach to temperature of 40 ^{0}C using water bath. After it reaches to 40 ^{0}C the sample was poured in to the viscometer and the reading from the display was taken as its viscosity value.

2.2.4.5 Pour point

The specimen is cooled inside a cooling bath to allow the formation of paraffin wax crystals. For every 3 °C after, the test jar is removed and tilted to check for surface movement. When the specimen does not flow when tilted, the jar is held horizontally for 5 seconds. If it does not flow, 3 °C is added to the corresponding temperature, since this is the last measurement when flow was observed, and the result is the pour point temperature [17].

2.2.5 Experimental Design for methyl ester Production

In these work the methyl ester was prepared using purified castor oil and methanol with a homogeneous catalyst of potassium hydroxide.

The transesterification process variables studied were molar ratio of methanol to oil and weight percentage of catalyst, reaction period, temperature and rotation speed was set at optimum point where the maximum conversion could be achieved based on literature data. Atmospheric pressure was used for all the runs.

2.2.5.1 Experimental Setup

A 250 ml glass reactor equipped with mechanical stirrer, thermostat, and condenser was used in all experiments. The reactor was connected to a water bath thermostat which was capable of controlling the temperature within deviation of 1^0C. A mechanical stirrer fitted with stainless steel propeller provided the mixing requirement.

2.2.5.2 Methyl ester Production Procedure

About 50 ml of castor seed oil was poured into a 250 ml glass reactor. The reactor assembly was then heated to temperature of 65 ^{o}C, 14.627ml methanol and 0.4775 gmhomogeneous catalyst was added to the reactor. The reaction was preceded for 1 hr.

2.2.5.3 Separation of glycerol from methyl Easter

Finally, after transesterification was carried out, catalyst and glycerol part was separated from the methyl Easter mixture by separatory funnel. Then, unreacted methanol and trace moisture was removed in oven for 5 hour. The methyl ester was obtained as a clear amber-yellow liquid.

2.2.6 Blending of methyl ester with additives

SLES and Cocamidopropylbetainewere used in order to improve its characteristic. For this process 10% SLES and 4% cocamidopropylbetainewere mixed with fatty acid methyl ester. This mixture was blended until foam formation stabilizes and the additives completely solubilize in the ester by continuously supplying heat.

2.2.7 Characterization of bio lubricant

The synthesized lubricant needs to remove the carbon residue deposited on surface of material. In order to check this property of the lubricant, the sample was taken to clean carbon deposited which was accumulated on magnetic stirrer.

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III. Results and Discussion

3.1 Seed Preparation, Oil Extraction, Oil Purification and Oil Characterization

3.1.1 Seed preparation

For 8.602 kg of castor seeds 0.602 kg husk was carefully removed. After removing the husks and the wastes, 8kg of castor seed was obtained. From this the castor bean weight was 93 % of raw seed weight.

3.1.2 Moisture content

The test of the castor seed results were summarized in table below.

Table 3.1 Determination of Moisture Content

Time (h)	0	2	6	8
Weight (g)	31.918	29.46	29.06	29.04

Therefore, the average moisture content of the bean was calculated to be 9.83%. According to the paper named "Physical and Nutrient Characterization of Raw and Processed Castor (*RicinuscommunisL.)* Seeds in Nigeria" [20], the average moisture content of castor seed was 7.25% which is a lower value as compared to the result of this paper. Little increment of moisture content could be due to the species of the castor and the prior washing of the seed before oil extraction.

3.1.3. Extracted Oil

From 150 g of purified seed, 37.5 ml of oil was achieved, then the density (ρ) of the crude oil was measured and the result was obtained to be 955 kg/m^3.From this 35.82 gm of the oil was extracted and the oil content (oil yield) of the seed was calculated to be 23.87 %.Consequently, its value was found to be below the previous findings [18].The low value of the oil content result was due to poor efficiency of the mechanical press and yield of the oil content of seed can be increased by improving the efficiency of the press.

3.1.4 Oil purification

The total amount of crude castor oil obtained from extraction was around 2 liter from 8 kg of castor seed. The crude castor oil was degummed to remove phosphatides, gums and other complex compounds in the crude oil using 3% (v/v) ml of distilled water which was 60ml. After centrifuge 1.8 liter of oil was obtained.

3.2. Physicochemical properties of castor seed oil

The density, kinematic viscosity, acid value, free fatty acid composition and moisture content of purified castor oil was determined and the results were given in Table 3.2.

Table 3.2: Physicochemical properties of purified castor oil

Property	Experimental Result
Density at 22℃(g/ml)	0.955
Kinematic viscosity at 40℃ (mm^2/s)	218.18
Acid Value (mg NaOH/g oil)	2
Composition of Free Fatty Acid (%wt.)	1
Moisture content (%wt.)	0.262

As stated on paper which was about Synthesis and characterization of biodiesel from castor bean as alternative fuel for dieselengine [8]. The density obtained for castor oil at 15℃ was 961.8 kg/m3 which could be approximated equal with the value gained in the paper synthesis and characterization of biodiesel from castor bean by Birova and Cvengros [8].

The composition of Free Fatty Acid and moisture content of the oil was below the result reported by [19] which was 2% and 0.5% respectively. 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 Easter was simple which was going to minimize extra cost of production of bio lubricant.

3.3. Fatty acid methyl Easter physicochemical Properties

The density measurement of the fatty acid methyl ester produced was performed and observed to be 888.92 kg/m^3. When it was compared to the EN 14214 standard for methyl ester which is 860- 900 kg/m^3, and the result was acceptable.

The dynamic viscosity of fatty acid methyl ester at 20.7^{0}C was measured to be 5.95 m.pa.sec and 3.42 m.pa.sec at 40^{0}C. This has been resulted in the kinematic viscosity of 6.69 mm^2/s at 20.7^{0}C and 3.847 mm^2/s at 40^{0}C. According to EN 14214 standards the kinematic viscosity of fatty acid methyl ester is 3.5-5.0 mm^2/s which made the result to be acceptable.

Table 3.3.Physiochemical properties of bio lubricant

Property	castor crude oil	castor bio lubricant	ISO VG-46	Petroleum based lubricant*
Density@ 25°C (Kg/m3)	955	888	_	885.6
Viscosity@ 40°C (cSt)	218.18	30.4	>41.4	10.801
Pour point		-5	-10	-9

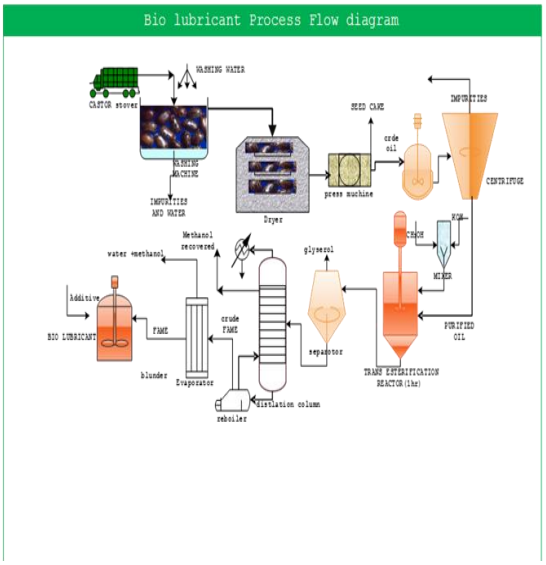

Fig.1. Process flow diagram of bio lubricant production

The synthesized lubricant removed the carbon residue deposited on surface of material comparable with petroleum based lubricant which is available in the market of the country. But its viscosity without any additional composition it didn't much with the standard of lubricants. This issue could be resolved by viscosity builder additive as like petroleum.

IV. CONCLUSION

The characterization of extracted oil were results density 0.955 g/ml, viscosity 218.8 mm^2/s, moisture content 0.262%, Acid Value (mg NaOH/g oil) 2, Composition of Free Fatty Acid (%wt.) 1 and oil yield 23.87%. Bio lubricant was produced using methanol alcohol and potassium hydroxide catalyst at constant reaction time for 1 hour at atmospheric pressure. The transesterification reaction was done at a temperature of 65^oC, 1.0% (w/w) KOH catalyst amount and for 7:1 molar ratio of alcohol to oil. The yield of this reaction was 98%. After the synthesis of methyl ester, formulation of bio lubricant was done by blending it with some additives like SLES and cocamidopropylbetaine. The physicochemical properties of the bio lubricant were, density 888 kg/m^3, viscosity 30.4 mm^2/s, and pour point -5℃ were obtained.

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