TRIBOLOGICAL EVALUATION OF LUBRICANTS DEVELOPED FROM SELECTED VEGETABLE BASED OILS FOR INDUSTRIAL APPLICATIONS

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ABSTRACT

Mineral oil based lubricants are non-renewable, harmful to health and prone to price fluctuations. Thus, vegetable oils are considered as suitable alternatives to mineral oils for lubricant production. Research on the use of non-edible vegetable oils for lubricant development has become necessary. The tribological evaluation of lubricant developed from non-edible vegetable (jatropha and castor) oils for industrial applications have been carried out in this study. The oils were characterized, modified for suitability and used to develop lubricants for industrial applications. Commercially available mineral oil based lubricant SAE 20/W50 was used as a control. The effect of additives on the tribological performance of jatropha and castor oil based biolubricants developed were also studied. Whereas modification of jatropha oil improved its viscosity but reduced its viscosity index, modification of castor oil greatly reduced its viscosity but improved its viscosity index. Jatropha and castor oils unlike other vegetable oils, possess excellent cold flow properties and modification of the oils further enhanced their cold flow properties. The developed jatropha and castor oil based bioluricants had alkaline pH, high viscosity index, appreciable viscosity, excellent cold flow and corrosion inhibition properties and highly biodegradable with a biodegradability exceeding 80 %, while the mineral oil based lubricant SAE 20/W50 had a poor biodegradability of 35.2 %. The extreme pressure additive cetyl chloride had the most significant effect on the coefficient of friction and anti-wear additive Tricysl Phosphate had the most influential effect on the viscosity index of the jatropha and castor oil based biolubricants. The jatropha and castor oils performed better in friction reduction and wear prevention than the SAE 20W/50 which had 0.114 coeficient of friction and 0.0067 $mm^3N^{-1}m^{-1}$ wear rate. The castor oil had 0.082 coefficient of friction and 0.007 $mm^3N^{-1}m^{-1}$wear rate. The developed jatropha oil biolubricant had coefficient of friction of 0.075 and wear rate of 0.00699 $mm^3N^{-1}m^{-1}$ and was better in friction performance but had similar wear performance with SAE 20W/50. The developed castor oil based biolubricant had 0.067 coefficient of friction and 0.00511 $mm^3N^{-1}m^{-1}$ wear rate and exceeded the SAE 20W/50 in friction and wear performance. Thus, modified and unmodified jatropha and castor oils have been found to be suitable for industrial applications in systems exposed to low temperatures. The developed jatropha and castor oil biolubricants have been found to be suitable environmentally friendly substitutes to mineral oil base lubricant SAE 20/W50 for application in two stroke engines, metal cutting and lubrication of gears in food processing industry.

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ABBREVIATIONS, GLOSSARIES AND SYMBOLS

A	Anti-wear Additive
Al	Aluminum
ANOVA	Analysis of variance
AOCS	American Oil Chemist Society
ASTM	American Society for Testing and Materials
AW	Anti-wear
B	Viscosity Modifier
C	Extreme Pressure Additive
CBL	Castor biolubricant
CO	Castor oil
Cu	Copper
D	Anti-Corrosion Additive
DSC	Differential Scanning calorimetry
ECO	Esterified castor oil (modified castor oil)
EJO	Esterified jatropha oil (modified jatropha oil)
EP	Extreme pressure
EU	European Union
FFA	Free fatty acid
FTIR	Fourier transform infrared
FTP	Flash temperature parameter
GC-MS	Gas chromatography-mass spectroscopy
GLWQI	Great lakes water quality initiative
HEC	Hydroxy ethyl cellulose
IV	Iodine value

JBL	Jatropha biolubricant
JO	Jatropha oil
KOH	Potassium hydroxide
LD50	Lethal dose for 50% of the population (measure of toxicity)
Mg	Magnesium
MNL	Mineral oil based lubricant (SAE 20/W50)
MOA	Multi-element oil analyser
N	Newton
OFAT	One factor at a time
PAHs	Polycyclic aromatic hydrocarbons
RBOT	Rotary bomb oxidation test
SAE	Society of Automotive Engineers
Si	Silicon
S/N	Signal to noise ratio
TCP	Tricrysl Phosphate
TGA	Thermogravimetric analysis
TMP	Trimethylolpropane
U.S. EPA	United States Environmental Protection Agency
VII	Viscosity index improver
WAFT	Water Aquaculture and Fisheries
ZDDP	Zinc dialkyldithiophosphate
ZnDDC	Zinc diakyldithio carbamate

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CHAPTER ONE

1.0 INTRODUCTION

1.1 Background to the Study

Tribology is the science and technology of bodies touching each other while moving; it includes the study of friction, wear and lubrication (Nosonovsky, 2010). Friction and wear come from complicated and multi-complex communication among bodies moving while touching each other. Every moving, observable, and shape aspects of the communicating bodies and their neighbourhood influence the surface communication and consequently the tribological behaviour of the entire arrangement.

Also, it can be seen from the systems aspect of friction and wear that the modelling of friction and wear is very difficult. There is dearth of good models that describes comprehensively the friction and wear process; thus, the interpretation of friction and wear data becomes complicated. Additionally, there is no correlation between friction and wear for example low friction does not automatically mean low wear rate (Menezes *et al*., 2013).

Wear is the continual removal of material from the surface in sliding or rolling contact against a counter surface. Based on the mechanism of material removal we have the following types of wear; adhesive wear, abrasive wear, wear caused by surface fatigue and wear due to tribochemical reactions. Over a longer sliding distance, either one mechanism alone or a combination of several of these wear mechanisms, causes a continual removal of material from the mating surfaces. Continuous steady-state wear and friction conditions may be quantified in terms of wear rate, which is mass or volume of material removed per sliding distance or time. Wear is the main cause of material wastage and loss of mechanical performance therefore; any reduction in wear

can give rise to considerable savings (Stachowiak and Batchelor, 2000). The major cause of wear and energy dissipation is friction.

Friction is the opposition experienced when two bodies touching each other are moving simultaneously, and it results to energy dissipation at the sliding interface. The dimensionless material property calculated as ratio of tangential force to normal load is called coefficient of friction. Normal load and frictional force are directly proportional to each other; therefore, friction coefficient is constant. It is assumed about 33.33% of the global energy resources is wasted to overcome friction in one form or the other. Reasonable amount of money can be saved via improved friction control and oiling or use of grease, which is an effective means of achieving wear and friction reduction (Bhushan, 2013a).

Lubrication is defined as the process or technique of using a lubricant to reduce friction and/or wear in a contact between two surfaces. Tiny layers of little tearing strength gas, liquid or solid are introduced among two surfaces so as to better the smoothness of movement of one body over another and thereby hindering damage. These thin portions of matter that separate contacting solid objects are often difficult to observe. Mostly, the width or height of these thin portions of matter are between 1 – 100 micro meters, although wider and higher films can be encountered.

The act of making better and or investigating useful influence of tiny films in hindering damage in solid-solid contacts is commonly termed lubrication (Bhushan, 2013a). Lubricants minimize friction, prevent wear, provide cooling and transport debris away from interface. Lubricants can be solids, solid/liquid thick colloid such as grease, liquids, liquid-liquid dispersions or gases. A thin layer of gas is suitable for low body to body force while solid thin layers mostly apply to low velocity bodies

communication. Detailed observation of fluid thin layers is usually called ‘hydrodynamic oiling’ while ‘solid greasing’ is the one done by solids. A specialized form of liquid or gas oiling where face to face interaction among the communicating objects and the liquid or gas smoothener is referred to as ‘elastohydrodynamic smoothening’ which is of beneficial applications. Smoothening that involves the chemical reactions between the liquid lubricant and the surfaces being lubricated is known as ‘boundary and extreme pressure lubrication’. Significant factors that affect lubricant effectiveness include:

1) rheological properties for example viscosity, viscosity index, pressure-viscosity Index.
2) Chemical properties for example reactivity with the top layer, boundary film-forming behaviours, abnormally high pressure constituents and shear force of solid lubricant or paintings.
3) Heat energy behaviours of smootheners for example specific heat, conductivity and diffusivity.
4) Hotness or coldness properties of oils for example pour-point temperature, cloud-point temperature, smoke point temperature, flame point temperature and resistance to oxidation.

Functionality of lubricants is defined by their chemical structure and their physical properties. The chemistry of hydrocarbon Base Lubricants is derived from organic chemistry. Different categories are given by their chemical composition and structure (Dresel & Mang, 2007).

Failure in machine parts due to absence or wrong choice of lubricants brings enormous cost (Dowson, 1979; Musa & Omisanya, 2021). In a 1986 survey it was revealed that supplying all the worm gear drives in the United States of America (USA) with a

lubricant that allows a relative increase of 5% in the mechanical efficiency compared to a conventional mineral oil would result in savings of about US$ 0.6 billion per annum (Pacholke & Marshek, 1986). The total cost of energy waste and material loss resulting from frication and wear is approximately $40 billion a year for the various industries in the USA (Jost, 1981; Stachowik & Batchelor, 2000).

Lubricants are very important consumables in virtually every industry. Within the last decade the annual worldwide consumption of lubricants is over 40 million metric tonnes. According to Ajithkumar (2009), lubricants are made up of over 9 out of 10 parts base oil and lower than 10% package of additive. The base oil used for the formulation of most lubricants is environmentally hostile mineral oil and 30% of lubricants consumed ends up in the ecosystem (Bartz, 2006; Ajithkumar, 2009).

However, mineral oil reserve is depleting and the environmental concern about the damaging impact of mineral oil is growing. The search for environmentally friendly substitutes to mineral oils as base oils in lubricants has become a frontier area of research in the lubricant industry. Vegetable oils are perceived to be alternatives to mineral oils for lubricant base oils due to certain inherent technical properties and their ability to be biodegradable.

Plant base oils when placed side by side with petroleum oils, has large fire point, elevated temperature rheology stability, high oilness and loss due to evaporation (Adhvaryu & Erhan, 2002, Mercurio, *et al.*, 2004; Souza, *et al.,* 2019). Vegetable oils have been found to be of less hazard to the ecosystem during deliberate or accidental release into the environment compared to mineral oil (Mercurio *et al.,* 2004; Awoyale *et al*., 2011). Poor hydro-oxidative homogeneity, large hotness or coldness sensitivity of wear and friction behaviour and poor low temperature behaviour are acknowledged

to be the hindrance of plant oils application as parent material for smoothness oils used in industries (Erhan & Asadaukas, 2000; Adhvaryu *et al.,* 2005). The present labours to eliminate these problems includes the addition of chemicals, transformations using heat and chemicals (Li & Wang, 2015). This work aims at developing lubricant from non-edible vegetable (jatropha and castor) oils and to evaluate the tribological properties of the developed lubricants. The research also studied four selected additives to determine which of the additives has the most significant effect on the tribological performance of lubricants developed from jatropha and castor oils.

1.2 Statement of the Research Problem

Wear and tear due to friction in moving contacting machines parts is a major problem in most industries. Lubricant has been developed to mitigate this negative effect of friction, commercially available lubricants are made from mineral oil which is harmful to the environment and humans. Besides this, petroleum reserve is depleting globally leading to increase in the cost of mineral oil available for lubricant production. These problems call for attention to search for cheap available alternatives that have no controversy for food security and non-edible vegetable oil has been identified as a viable option.

Several researches have been carried out that studied the development and performance of bio-based lubricants for industrial applications. However, there are scanty literature on the tribological performance of non-edible vegetable oil based lubricants. Specifically, there are limited publications on the tribological properties of lubricants developed from jatropha and castor oil. Research on the friction and wear performance of lubricants developed from jatropha and castor oils is an area needing attention and this research seeks to address this gap.

The overall tribological performance of a lubricant is a function of the base oil and additives. There are very few existing literatures on the influence of the concentration of additives on the wear and friction performance of jatropha and castor oils. This research will also consider the determination of how the tribological performance of lubricants developed from jatropha and castor oils will be affected by additives.

1.3 Aim and Objectives of the Study

The aim of this study is to evaluate the tribological performance of lubricants developed from selected vegetable based oils for industrial applications. The aim will be achieved through the following objectives:

1) Determine and modify the profile of jatropha and castor oils for lubricant production.
2) Develop lubricant from modified jatropha and castor oils.
3) Evaluate the tribological properties of lubricant developed from modified and unmodified jatropha and castor oils.
4) Determine the effects of additives on the tribological performance of the developed jatropha and castor oil based biolubricants.

1.4 Justification of the Study

Tribological deficiencies which are largely due to inappropriate use of lubricants or the absence of lubricants produces enamours losses (energy and material) that occurs simultaneously in all mechanical devices in operation. Use of mineral based lubricants is harmful to environment and health of humans involved in the production and use of mineral based lubricants. Therefore, this research seeks to develop environmentally friendly and non-harmful, biodegradable lubricants from two vegetable oils that are produced in Nigeria.

The use of edible vegetable oil for lubricants formulation is challenging as there is competition for its use as food. This work developed industrial lubricant from non-edible vegetable (jatropha and castor) oils. This research is therefore a step further in the right direction to solve the food versus debate on the use of vegetable oil.

This research will encourage rural people to use waste and non- arable lands for non - edible oil production which will reduce poverty and create jobs among the poor. The reduction in energy and material losses that is envisaged to be derived from the outcome of this research is most significant in this period of global economic down-turn.

Despite the progress in research in the use of vegetable oils for the development of industrial lubricants, literature is scanty on the tribological performance of jatropha and castor oils. Additionally, there is a dearth of knowledge on the tribological evaluation of lubricants developed from modified castor and jatropha oils.

Most of the additives used for the formulation of lubricants contain heavy metals as well as sulphur and phosphorous compounds that are harmful to man, animals and the environment; there is therefore need to search for minimum quantity of additive to be used for vegetable oil based lubricants formulation and this work is a further effort in search of which selected additives most significantly affects the tribological performance of developed lubricants. The knowledge of the most significant additive will aid in reducing the number of additives to be used thereby protecting the environment from harmful chemicals.

1.5 Scope of the Study

This work evaluated the density, flash point, pour point, viscosity, viscosity index, corrosion resistance, oxidative and thermal stability of locally available jatropha and

castor oils. Chemical modification of the jatropha and castor oil were carried out followed by the development of lubricants from the modified vegetable oils. Evaluation of the tribological performance of the lubricants developed from the modified and unmodified vegetable oils was also carried out. The work also determined the most significant additive that affects the tribological performance of the developed lubricants using design of experiment through the Taguchi method. Only four selected environmentally friendly additive were used in this study and the tribological performance of the developed lubricant was evaluated using ball-on-disc tribometer only.

CHAPTER TWO

2.0 LITERATURE REVIEW

2.1 Theoretical Fundamentals

Tribology involves friction, wear and lubrication. The theoretical background of this research is presented in the following sub-sections.

2.1.1 Lubricants for industrial applications

Materials interposed among moving members to bring down wear and friction, radiate heat, flush out contaminants, and better efficiency are called lubes (Menezes *et al*., 2013). The exact formulation of a lubricant depends on the particular use or desired application marked out by the manufacturer. Lubricants distinquish themselves from each other as a result of the diverse range of functionality of a lubricant.

Tribological system (or tribosystem) consists of four components, namely: (i) a contacting surface, (ii) an opposing contacting surface, (iii) the contacting interface along with the lubricant medium in the interface, and (iv) the environment and all external properties (Mang & Dresel, 2006). A lubricant function by introducing a medium with lower shear strength than the opposing surfaces. In some lubricated tribosystems, the lubricant may not completely prevent asperity contact between the surfaces. However, the lubricant will reduce the number of asperity contacts and it may also reduce the shear strength of the junctions formed during asperity contact. In other cases, the lubricant completely separates the surfaces and no asperity contacts are formed at all.

Lubrication regimes are normally associated with dominant lubrication mechanisms involved in the mechanical system as illustrated in the Stribeck curve for liquid

lubrication shown in Figure 2.1. There are three main lubrication regimes: boundary lubrication, mixed/elastohydrodynamic lubrication, and hydrodynamic lubrication.

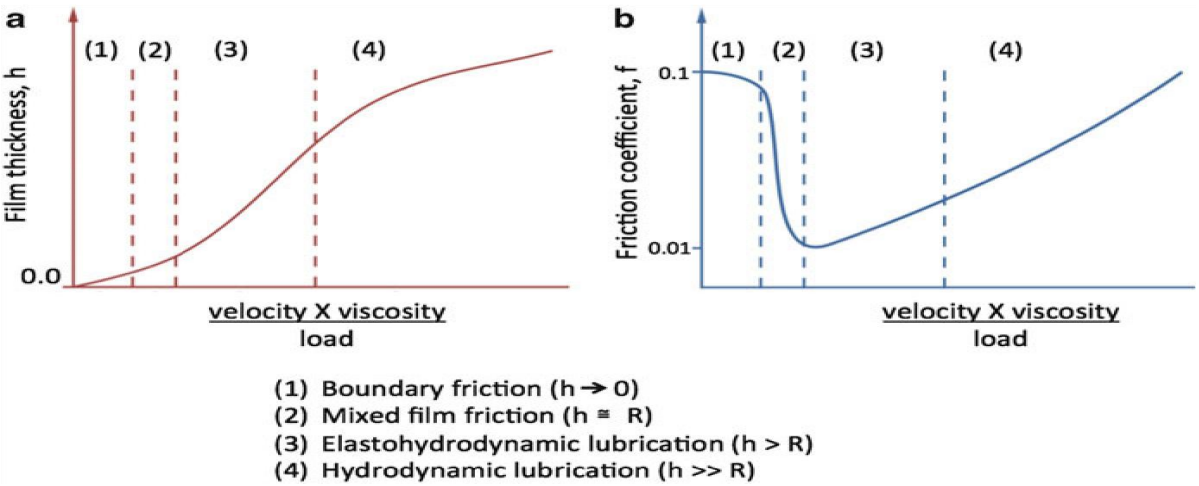


Figure 2.1: Demarcation of the four sections of lubrication: (a) thin layer thickness vs. Hersey's number and (b) friction coefficient vs. Hersey's number (Stribeck curve) (Menezes *et al,* 2013).

Considering where oils for wear and friction control are sold, a wide range of uses directly created for lubricants have produced more than 10,000 kinds of oils that meet the needs of more than 90 % of all lubricant applications globally (Mang & Dresel, 2006). Figure 2.2 shows the worldwide lubrication market as early as 2004. It is counted that 37,400 million kilograms of lubricant is being consumed (Mang & Dresel, 2006). Transport and industrial lubricants are the most prevalent in the lubrication market.

Industrial lubricants took up 8/25 parts and were made up of 3/25 parts hydraulic oils, 1/10 parts other industrial oils, 1/20 parts metalworking fluids, 3 out of 100 parts greases, and 1/50 parts industrial gear oils (Bartz, 2006; Lingg & Gosalia, 2008). The 1

out of 10 parts industry oils labelled industrial lubricants is made up of fluid compressor oils, bearing and circulating system oils, air conditioning compressor oils, and turbine oils. In the automobile area, the frequently used fluid lubricants were petrol and diesel engine oils, automatic power transfer fluids, gearbox oils, brake fluids, and hydraulic fluids. Everywhere, three types of lubricating substances exist: liquid, solid, and gaseous lubricants.

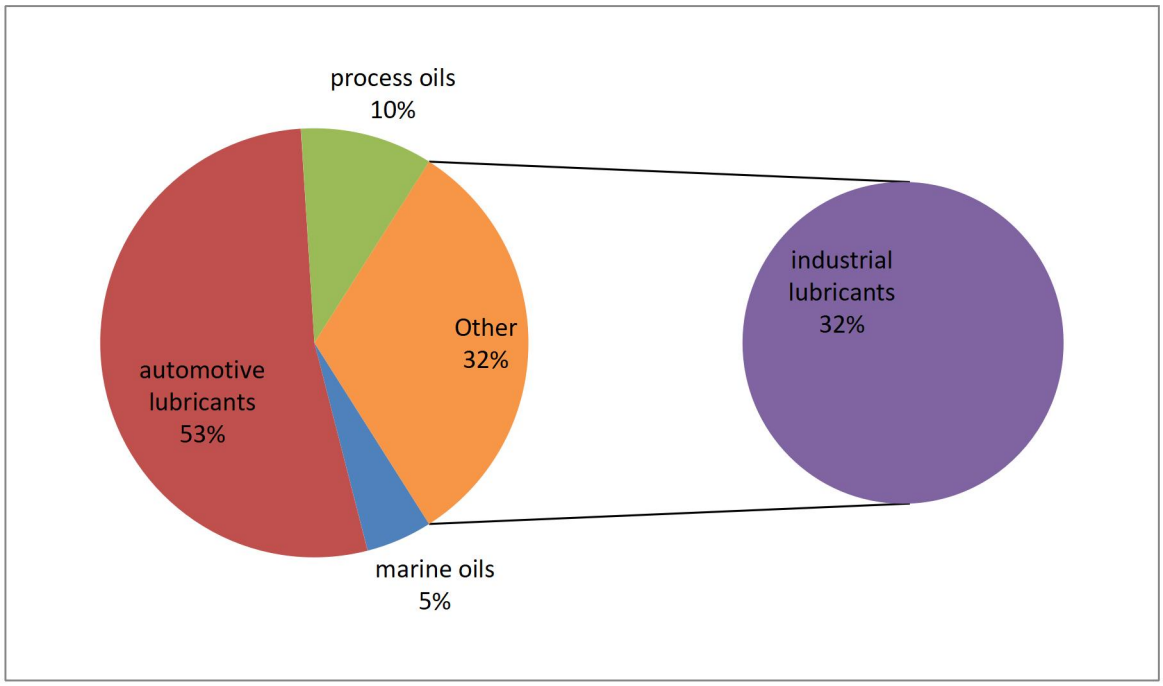


Figure 2.2: Worldwide lubrication consumption in 2004 (Mang and Dresel, 2006)

i. Liquid lubricants

Liquid lubricants are evaluated in many kinds of procedures. Commonly used procedure has to do with the type of parent material oil involved. According to Bhushan (2001), recognised parent material oil types are water-repellent lanolin, water, petroleum oils, oils from living things, and artificially created oils (made up of non-petroleum oleo-chemical synthesized compounds). Incompressible fluid lubricants are high viscosity fluids that needs pumps or rotating systems like gears or bearings to supply the lubricant to every machine member (Galgat *et al.,* 2021).

The basic use of a water-like lubricant has to do with limiting wear, resistance to motion, and surface damage throughout the designed system life time. Other functions of fluid oils include prevention of corrosion and removal of heat, unwanted materials, and worn out particles (Galgat *et al.,* 2021). During other applications like in hydraulic systems, fluid lubricants are used for either force or power transmission. The major limitations of fluid lubricants are the depreciation in load carrying ability during very hot or very cold operations and degradation while in use. Thus, the service provided by the liquid lubricant is predetermined by its makeup and its observable and reactive properties.

ii. Solid lubricants

Solid or non-fluid smoothening agents are generally powder or grease which are quasi-solids or solid–liquid suspension. Graphite (C), molybdenum disulphide (MoS_2), Tungsten disulphide (WS_2), and Titanium dioxide (TiO_2) are the predominantly available powder lubricant materials (Menezes *et al.,* 2013). Solid lubricants offer lubrication at temperatures (around 350^0C) that are higher than many liquid oil-based lubricants. Solid lubricants such as Teflon or polytetrafluoroethylene (PTFE) are specifically used as a surface coating layer to prevent sticking of the contacting surfaces (Blanchet & Kennedy, 1992).

Solid–liquid suspensions consist of solid lubricants mixed into a liquid fluid such as water or oil to form a colloidal suspension. These slurry or two-phase lubricants are often utilized in metalworking processes such as forging and extrusion. Solid lubricants can also be used in a less viscous approach where they are applied as suspension in a carrier fluid such as vegetable oil (Reeves *et al.*, 2012). Greases are complex semisolid lubricants consisting of liquid-base oils mixed with various

thickening agents derived from soaps or other organic or inorganic substances. Greases can also include a multitude of additives such as powder lubricants and their consistency closely resemble that of a paste (Menezes *et al.*, 2013). They range in multiple viscosities and are available in semisolid (also called semiliquid) form to solid form known as block greases. Greases are often processed in special grease-making facilities, as they require special equipment for their production.

iii. Gaseous lubricants

Compressible fluid lubricants exhibit very small viscosity compared to incompressible fluid or densely packed lubricants. Gaseous lubricants offer less heat capacity and bigger compressibility than incompressible fluid or densely packed lubricants (Bhushan, 2001). Examples of compressible fluid lubricants are air, engineered gases, steam, and metals in liquid or gas form. These lubricants applied in special bearings, where circular motion forces enable air to force the foil and the shaft apart so that there is very slight touch. Another instance is in wind hockey tables, a reknown play at arcades, that employes reduced volume air to provide a non-thick air platform on which the puck glides over allowing for an almost zero friction space.

2.1.1.1 Lubricants and the environment

Lubricants are made up of oils that are dangerous to humans, plants and animals due to their tendency to slip into and poison all forms of water bodies. Over the last decade, the ecological impact of lubricants has become a growing concern. Such concern include; emissions toxicity, water hazard potential, and biodegradability. Toxicity is the chief property of lubricants when evaluating the chances of poisoning to humans and other living things. Waste lubricating oils are dominated by cancer causing four rings polycyclic aromatic hydrocarbons (PAHs).

Biodegradability studies of lubricants having plant and animal origin were also carried out by Luna *et al.* (2015) using a bio-kinetic model. The result established that the pure castor oil was most biodegradable with half-life of 12 days, followed by biolubricants from castor oil with half- life between 20 to 30 days; but mineral oil had half-life of over 200 days. It was also discovered that there was no much change in biodgrability of lubricants developed by esterification of castor oil using different kinds of alcohols.

Bartz, (1998) and Bartz, (2006), reported that any material that satisfy the following consideration will be an eco-friendly lubricant.

1) The material should be chemically unreactive with the surroundings as at when produced; take in little power; generate no waste nor emission.
2) If the material is gotten from replenishable source to prevent it from running out of supply and avoid it from adding dangerous gases into the atmosphere.
3) Medically the material is safe and not having tendency to cause cancer nor be a poison.
4) Not produce harmful by products when broken down; neither pile up biologically harmful products.
5) The material is ecologically and medically acceptable; without hazard to the hydrosphere and be able to float in water.
6) The lubricants should have high rate of biodegradability and should not emit any harmful or unwanted by-products when broken down.
7) The lubricant should not be difficult to dispose; should be easily recyclable.

Aquatic Species	Concentration (mg/L)	Time (HR) to LC50
American Flagfish (jordanella floridac)	1 to 9.5	96
Fathead minnow (Pimephales promelas)	1 to 9.5	96
Daphnia magna	4.65	48
Clam (Mercenaria) embryo	0.04	48
Clam (Mercenaria) larvae	0.1	48

Table 2.1: Used lubricant toxic effects on aquatic organisms

(Source: Fan, 2010)

Waste lubricants are seriously dangerous to living organism both old and young. Other damaging consequences are dwarfness, gene mutations and higher death rate (Albers, 1980). Numerous researches affirmed that disolveable portions of waste lubricants are very harmful to creatures living in water bodies (Byrne & Calder, 1977*;* Hedtke & Puglisi, 1980; Silva *et al.,* 2013). Table 2.1 depicts poisonous effect of dissolvable parts of waste lubricants in water bodies. LC50 is the specified procedure to quantify poison level to show the deadly amount that will eliminate half of experimental organism over a specified period.

Non-animal organisms are also susceptible to waste lubricants attacks. Green leaves, like radishes, turnips, and beans studied, were planted on various soils (sandy loam and clay loam) upon which waste lubricants were later deposited on. The results showed no single of the green leafs survived at silt loam and sandy loam after nine months. The ones that survived on the poisoned clay loam, were dwarfed and sickly with yellow leaves and yellow stalk (Raymond *et al.,* 1976).

Grains like corn and sorghum are not spared from the effect of waste lubricants. Maize growth retarded by 46% on (1300 ml/m^2) poisoned soil. Sorghum growth also retarded by 76% by harvest time (Giddens, 1976). The same kind of experiment was carried on water bodies to measure the poisoning of the plant and animals. Bott and Rogenmuser (1978) and Bate and Crafford, (1985) discovered that the population of freshwater algae dramatically dropped, and the food production capacity per time of phytoplankton were opposed as well.

Sources of Water both on land and underneath land may be contaminated by used lubricant by the following means:

a) lawless dumping of expended lubricants in toilets;
b) combining expended lubricants and household dirts; and
c) leaks or accidental discharge of expended oils running-off into the hydrosphere.

Used lubricants illegally disposed into stormwater sewer systems can significantly pollute surface water and cause harmful effects to the aquatic ecosystems. Stormwater runoff carries used lubricant spills into sewers is another possible route for used lubricants getting into surface water. Used lubricant film may damage the water body and aquatic ecosystems by blocking sunlight for photosynthetic processes in water and impeding the cycle of dissolved oxygen (Fan, 2010). In addition to the oil film, metals like zinc, copper, and cadmium; and water soluble PAHs, such as benzopyrene, naphthalene, may dissolve into the water and cause considerable damages to aquatic organisms (Payne & Philips, 1985).

Expended lubricating matter may attach on organic dirt inside landfills when expended lubricants are discarded in combination with household rubbish (Fan, 2010). In

dumpsites with penetrate-able basement, dissolved portions of expended lubricants can easily permeate wastes eventually poisoning groundwater via leaching, runoff or percolation. Because of rapid development in industrialisation, the demand for lubricants is increasing and the commonly used base oil for lubricant formulation is mineral (petroleum) oil, as at 2005 only 1% of lubricants consumed were made from vegetable oil (Bartz, 2006). However environmental concern, fluctuating petroleum price and tight regulations about the use of environmentally friendly lubricants have given rise to renewed interest in the research of vegetable oil base lubricants.

2.1.2 Vegetable oils as lubricants

Vegetable oils have been used as lubricating oils from ancient days (Dowson, 1998). They are easily obtained from natural sources. Therefore, they had been the main ingredient of lubricating oils until the 19th century. The requirement of lubricants became very high thereafter because of rapid industrialisation, putting pressure on the price and availability of lubricants from vegetable and animal sources. Mineral oils were started being used as lubricating oils after the successful prospecting and extraction of mineral oils during the second half of 19th century which made available large quantities of cheap replacement for lubricants of vegetable and animal origin. Mineral oils provide various fluids which have desirable properties as lubricating oils at a reasonable cost. For that reason, most of the lubricating oils are supplied from petroleum-based materials.

Currently, request for eco-compartable oils is on the rise due to the high concern for environmental protection. Plant and animal based oils are renewable and, very biologically degradable, therefore, they are prospective materials for the parent oils for acceptable lubricants (Asadauskas *et al.*, 1996). Petroleum based lubricants were employed in all manner of industries since ancient times including automobile engines,

stamping and wire drawing applications and power transfer systems. However, not too long, it was discovered that petroleum based lubricants with the same rheology as bio-based oils was not as performing as the bio-based ones. Ratoi *et al.*, (2000) explained this to be a characteristic of the plant or animal based oils called "oiliness" or "lubricity". This characteristics of bio-based oils is taken to result from their ability to attach to iron-like material surfaces, giving rise to strong bond single-layer regions, where the positive head sticking to the metallic surfaces and the neutral chains pointing perpendicularly to the surface (Weijiu *et al.*, 2003).

Numerous additives are employed for boosting properties as well as shelf-life of lubricating oils. Various types of additives are being blended with lubricants, based on the requirement placed on the final product. Some of the additives widely used include soaps, dispelling agents, high-pressure performance (EP), wear-controling (AW), rheological index enhancers (VII), and corrosion preventers (Rizvi, 1999). The classical industrial lubricant is made up of parent oil and an property boosters package. Several nations like Austria, Canada, Hungary, Japan, Poland, Scandinavia, Switzerland, the USA, and EU have or are about making laws to control the use of petroleum based oil lubricants in delicate ecological regions (Bartz, 1998; Bartz, 2006).

The United States of America (U.S.A) market for all lubricants is 8,250,000 tons/year and only 25,000 tons/year were based on vegetable oils (Whitby, 2004). In the USA, executive order 12873 (EQ 12873) encourages the use of environmentally compatible oils where it is possible to meet the requirements. Similarly, the Great Lakes Water Quality Initiative (GLWQI) in the USA is intended to maintain, protect, and restore the unique Great Lakes resource (for example water quality). Within GLWQI, there are proposals to encourage use of fast biodegradable lubricating oils and limiting the use of potentially toxic (to aquatic life) additives to very low levels. In Austria use of

mineral-based lubricants, in particular applications like chain saw oils are banned. The European Community (EU) has released the Dangerous Substances Directive. It establishes criteria for a product's potential hazards to aquatic environment. This hazard potential is determined through assessment of aquatic toxicity, biodegradability, and bioaccumulation potential. The other countries mentioned above have at least established regulations to evaluate the lubricant caused impairment to the environment.

The set-backs in applying plant base lubricants is from the vegetable oils themselves rather than the additives blended with them. The parent oil makes up to 9 out of 10 parts of a lubricant and that is where the behaviour of the lubricant like high ability to breakdown naturally, low tendency to escape by evapouration, ideal cleanliness, high ability to dissolve lubricant additives, ease to mix-up with other types of system fluids, intangible attack on seals and elastomers, density and heat conductivity comes from (Erhan & Asadaukas, 2000). Parent oil materials prominently control resistance to oxidation, deposit forming ability, low temperature solidification, resistance to hydrolysis, and viscosity characteristics. According to Erhan and Asadaukas (2000), properties like oiliness, wear prevention, weld load, corrosion resistance, pH, ash content, colour, foaming, demulsibility and water repulsion rely totally on the additives or impurities/contaminants.

Therefore, when a given fluid is considered for its suitability as a lubricant, first of all, the base stock dependent parameters are evaluated. In addition to biodegradability, the following characteristics must be given attention: cleanliness (particle count), compatibility with mineral oil lubricants, homogeneity during long term storage, water content and acidity, viscosity, viscosity index, pour point, cloud point, cold storage, volatility, oxidative stability, elastomer compatibility and possibly other properties, depending on intended application. Water rejection, demulsibility, corrosion protection,

ash content and foaming could also be tested if contamination of the additive-free oil is suspected.

Technically, it might be argued that over 9 out of 10 parts of all current lubricants can be made to be highly biodegradable (Wagner *et al.*, 2001). However, there is still large room for further development work to be carried out and economic consideration is discouraging (Mang, 1997; Hill, 2000). Plant and animal oils including their derived esters are parent material for development of eco-friendly, fast biodegrading lubricants. Mang, (1997) and Van Voorst and Alam, (2006) reviewed and recorded formulation of environmentally acceptable, easily biodegradable lube oils using petrochemicals such as polyalphaolefins, polyglycols, polyalkylene glycols and artificial esters. Notwithstanding, renewable oil is chosen instead of these artificial fluids that are unsustainable and more expensive (Adhvaryu & Erhan, 2002).

Some of the rapidly biodegradable lubricants are based on pure, unmodified vegetable oils. In Europe, predominantly rapeseed oil and sunflower oil are used (Wagner *et al.*, 2001). Chemically, these are esters of glycerine and long-chain fatty acids (triglycerides). The alcohol component (glycerine) is the same in almost all vegetable oils. The fatty acid components are plant-specific and therefore variable. The fatty acids found in natural vegetable oils differ in chain length and number of double bonds. Besides, functional groups like hydroxyl groups as in castor oil may be present. Natural triglycerides are very rapidly biodegradable and are highly effective lubricants. However, their thermal, oxidative and hydrolytic stabilities are limited. Therefore, pure vegetable oils are only used in applications with low thermal stress. These include total loss applications like mold release and chain saw oils (Ajithkumar, 2009).

Despite plant oils exhibiting powerful oiliness at low temperatures, they are known to amplify material removal at elevated temperatures and in sliding conditions. Olive oil

and soybean oil produced high amount of wear when tested at 150ºC above a sliding speed of 0.4 m/s (Choi, *et al.,* 1997). Beyond certain threshold temperatures, fatty acid contents of plant oils exhibit large wear (Bowden & Tabor, 2001). The chemistry, type of fatty acid, sliding force and velocity and condition of the lubricated metals determines the transition temperature.

The two bonds in the fatty acid part and the "beta-Carbon-Hydrogen group" of the alcoholic (glycerine) components is responsible for the instability of plant oils in the presence of heat and oxygen (Wagner *et al.*, 2001). Specifically, technical application is limited by the presence of more than one bonds. According to Adhvaryu, *et al.*, (2005) the bis-allylic protons present in certain chains with many bonds are highly exposed to radical attack and later undergo degradation due to oxidation and form polar oxy-chemical products. That occurrence brings about higher pH and viscosity and solid deposits. Plant and animal oils additionally, when water contaminants are present display low corrosion protection. The beta-hydrogen particle is readily removed from the molecules, which tend to cleave to the esters and form other degrading by-products.

Another disadvantage of natural oils as reported by Goyan, *et al.*, (1998) is their hydrolytic instability when contaminated with water. Therefore, the presence of water in whatever form must be avoided at every stage. Bad cold flow properties include cloudiness, precipitation, poor flowability, and tendency to precipitate solids at elevated temperatures (Asadauskas *et al.*, 1996).

2.1.3 Modification of vegetable oil lubricants

Vegetable oils despite having superior lubricity compared to mineral oil, has some set-backs that limit their application as base oil for industrial lubricant application

(Ajithkumar, 2009). These limitations include the following; depressed rheology as compared to petroleum and artificial oils, poor oxidation stability, lower temperature range than that of petroleum and synthetic oils. Also, several of the characteristics change more with time than those of petroleum and artificial oils and low temperature properties are lower for vegetable oils than other lubricating oils.

Energy has been expended to better the cold flow properties via mixing the plant oils with diluents like multi α olefin, diisodecyl adipate, and oleates (Asadauskas & Erhan, 1999). Another achievable method for regulating these challenges is chemical transformation of the oils by organic reaction (Randles & Wright, 1992). Documented evidence shows that triacylglycerols that have complex organic structures possess less solidification temperatures (Ohkawa *et al.*, 1995; Rhodes *et al.*, 1995). Plant and animal based oils are usually categorised by their oil-chemistry fractions like fatty acids or fatty acid alkyl esters and glycerine before their transformation. Fatty alcohols are obtainable out of fatty acid alkyl esters. But natural oil can straitly be changed, for example, by direct organic neutralisation reaction or choice addition of hydrogen. The beneficially significant transformation involves the carboxyl group of the acid components of the oil. They account for 9 out of 10 parts of the oil-chemical transformations, whereas the acid chain reactions only give rise to less than 1 out of 10 parts (Kassfeldt & Goran, 1997; Rhee *et al.*, 1995).

2.1.4 Non-edible vegetable oil lubricants

Vegetable oil are either edible or non- edible in nature, various countries in the world import edible vegetable oil for food; for example, India has 16.6 million tonnes annual edible oil consumption and is the largest importer of edible oil (Jain & and Suhane, 2012). Nigeria is a net importer of edible oil. With increasing population and the inability to meet domestic demand for edible oil it is now very challenging to use

edible oil for lubricant formulation because of the food versus energy (lubricants and fuel) debate. Therefore, as alternative non- edible oil is gaining consideration for bio-lubricant and bio-diesel development. According to Ioan (2002) and Erhan *et al.,* (2006) non-edible vegetable oils besides being cheaper offer good or at least similar performance as mineral oil base in lubricants.

Production of lubricants from non-food oils will be a solution to the food versus energy, environmental and economic problems associated with the use of edible oil for lubricant and biodiesel production. Non-food vegetable oil plants are well adapted to arid and semi-arid conditions and requires little to no fertility and moisture. Moreover, non-food crops are considered to be planted on areas that are mostly depleted, located in poor locations and wasted forests thereby making biolubricants production from non-edible vegetable oil a major poverty alleviation programme. Locally available non edible oil includes Jatropha oil, castor oil, neem seed oil, rice bran oil and karanja oil. These plants can be propagated through seed or cuttings and their seed cake after extracting the oil can be used as fertilizer. There are three most commonly used methods of extraction of non-edible oils: mechanical extraction, solvent extraction and enzymatic extraction (Bilal *et al.,* 2013).

2.1.5 Jatropha oil lubricants

Jatropha curcas is a water filled short plant from the Euphorbiaceae family. The shrub came from Eastern part of Africa and is popular today in many nations throughout the temperate regions. Jatropha has good drought resilience properties, and can survive on nutrient depleted and sandy soils, except clay soil and swampy areas. This short plant protects land from erosion at high elevations. Jatropha is three years maturing tree, which survives for up to 40-50 years and can produce seeds for 25 years (Jain &

Suhane, 2012). It can grow in nutrient depleted lands, and does not compete for the food production with human or for space used in food production.

Jatropha oil is a non-food plant oil gotten out of the seeds of jatropha curcas seed. The plant is one of the most investigated oils for all forms of lubrication (Ho *et al.,* 2020). Its excellent viscometric properties is as a result of the prevailing ricinoleic acids, around 9 out of every 10 parts (Silva *et al.,* 2013), and high oleic acid (43%) suggests it can be used as a lubricant.

The eco-friendly lubricants gotten from Jatropha curcas oil have better cooling properties than that from palm oil, better rheology than that from castor oil and superior resistance to oxidation and better pH than that from soybean oil (Arbian & Salimon, 2010). The global annual production of jatropha oil is1.5 Mt. Africa has a contribution of around 1.08Mt. Jatropha plant is suitable for cultivation in several of the parts of Nigeria. The output of jatropha curcas seed in Nigeria is summed up at about 9,901,700 kg/ha (Mamuda *et al.,* 2016a), and all regions of Nigeria is good for jatropha farming. Mean oil quantity from jatropha seed considering the weight of the dry material is 58% (Nayak & Patel, 2010).

2.1.6 Additives

Additives can be defined as chemicals added to base oils to increase some properties of the resultant complex such that they can meet the increasing demand being made on them and meet certain specification. All base oils lack ability to fit-into the demands of a highly performing lubricant by themselves (Reeves *et al*., 2012; Garba *et al.,* 2019). Therefore, these chemicals are added to oils to extend or adjust properties of a lubricant. Existing properties can be extended, undesirable characteristics suppressed, and new properties introduce to the base oils by additives.

Predominant chemicals (additives) used in the formulation of biolubricants contain heavy metals like zinc, antimony and other harmful substances like sulphur and phosphorous. Currently some researchers are also turning attention to the development of environmentally friendly additives for biolubricant development.

2.1.6.1 Viscosity index (VI) improvers

Index of viscosity stabilizers refers to chemicals or natural substances which controls the decrease in oil viscosity when the oil is heated up. They are made of heavy chemical mass polymers, that are oil soluble. The polymers are chain-like molecules with solubility depending on chain length, organic shape, and material make up. The class of polymers that can impact these effects include hydrogenated styrene butadiene polymers, alkacrylate polymers, acrylate polymers, and olefin polymers (Audibert, 2006). According to Menezes *et al.* (2013), the quantity of viscosity controlling additives in lubricants lies between 3 and 30 % mass fractions.

2.1.6.2 Anti-corrosion additive

Chemicals that control corrosion (additives) are separated into corrosion preventers and rust preventers. Corrosion preventers usually protect nonferrous metal (aluminium, tin, cadmium and copper) materials against the attack of corroders in the lubricant. Rust preventers are employed to deliver iron and its alloys from corroders (Menezes *et al.*, 2013; Bhushan, 2013b).

2.1.6.3 Extreme pressure (EP) additives

Compounds formulated to combine with metal surfaces under abnormally high load and heat situations to prevent fusion of mechanical members are called extreme pressure (EP) additives. Closely related to anti-wear additives, EP attach onto the metal by strong bonding forces or chemical-adsorption due to their polar molecules.

Most times EP additives are made up of an aggressive non-metal, like sulfur, antimony, iodide or chloride molecules.

2.1.6.4 Anti-wear additives

Anti-wears form protective thin layer upon metal parts to prevent those metals from destruction. The additives include polar organic molecules (for example, alkanols), esters of fat, amines of fat, and organic chemicals containing sulphur, phosphorus, chlorine, nitrogen, lead, or zinc. Like EPs they function through chemical-adsorption reactions with host surfaces to produce tribological and chemical layers, that protects durable material surface layers. Compounds, such as Zinc diakyldithiophosphates (ZDDP), are deployed as both antioxidant additive and anti-wear additive (Mortier and Orszulik, 1997; Audibert, 2006). There are numerous kinds of anti-wear additives that presently are in use in oil blendings typically in concentrations of 1–3% by mass (Menezes, *et al*., 2013).

2.1.7 Taguchi Method of Design of Experiment

Design of experiment (DOE) refers to the process of planning experiments so that appropriate data can be analysed by statistical methods, resulting in valid and objective conclusions (Masounave *et al.*, 1997). Experimental design methods such as factorial design, Taguchi design and response surface methodology (RSM) are now widely used in place of one-factor-at-a-time (OFAT) experimental approach which is deemed time consuming and exorbitant in cost (Das *et al.,*2013). Design of experiments has been used to study the effects of machining parameters such as cutting speed, feed rate, depth of cut, tool nose and others, in order to determine such response as surface roughness, cutting temperature, and so on (Makadia & Nanavati, 2013). Experimental design is a critically important tool in the scientific and engineering world for improving the realisation process. Experimental design methods are also of

fundamental importance in engineering design activities, where new products are developed and existing ones improved. Statistical design of experiment refers to the process of planning the experiment so that appropriate data will be collected and analysed using statistical methods, resulting in valid and objective conclusions. There are two aspects to any experiment; the design of experiment and the statistical analysis of data.

Taguchi's method that is orthogonal in array is a data processing method under design of experiments which brings down repetitions of experimental trials and give sufficient information about the effect of control factors (Taguchi, 1986; Houng *et al.*, 2006). Various control factors can be investigated simultaneously and the result gotten with optimum significant values deduced using the Taguchi technique. Taguchi method created orthogonal array to observe the effect of control factors involved and to reduce experiment numbers. The experimental results obtained are used to calculate signal to noise ratio (S/N). The S/N analysis the quality properties to understand if the outcomes are diverging from or converging to the obtained results. There are three categories involved for quality characteristics to analyse S/N ratio which include: the smaller the better; the larger the better; and the nominal the better. These can be calculated according to the following equations (2.1) – (2.3):

The-smaller-the-better:

$$\frac{\mathrm{S}}{\mathrm{N}} = -10\log\frac{1}{n}(\sum_{i=1}^{n} y^2) \tag{2.1}$$

Such that n indicates number of repetitions and y is the outcome factor value. This is deployed where smaller value is required.

The-nominal-the-better:

$$\frac{S}{N} = 10\log(\frac{\mu^2}{\sigma^2}) \tag{2.2}$$

Such that μ stands for mean and σ stands for the variance. It is deployed where nominal or variation is minimum.

The-larger-the-better:

$$\frac{S}{N} = -10\log\frac{1}{n}\left(\sum_i^n \frac{1}{y^2}\right) \quad (2.3)$$

Such that n indicates number of replications and y is the observed response value.

2.2 Review of Past Works

2.2.1 Vegetable oil as universal tractor transmission lubricant

Stojilković and Kolb (2016) carried out a study on the tribological properties of biodegradable universal tractor transmission oil by using three vegetable oils: rapeseed, soybean and sunflower oil, by comparing their properties with a commercial mineral oil based universal tractor transmission oil. Tribological tests for all four samples were conducted on annealed alloy steel 16MnCr5 (C4320) having the hardness of 35 HRC. Block on disc tribometer TPD-93, with a sliding speed in the contact zone of 0.8 m/s and contact duration of 60 minutes. It was realised that the coefficient of friction of tested plant-based oils samples was smaller than that of the reference petroleum oil universal tractor transmission oil, especially at higher loads. Also, sunflower oil had the lowest wear scar width of 1.345 millimetres while the mineral based oil had the highest wear scar width of 1.585 millimetres. From the study it was concluded that rapeseed oil had the best performance with regards to coefficient of friction while sunflower oil performed best in terms of wear scar width, thus all the vegetable oils are better in tribological performance compared to the universal tractor transmission oil. All of the vegetable oils are edible and will there by further compound the problem of scarcity of available edible oil.

2.2.2 Vegetable oil as multi-cylinder engine lubricant

Kalhapure *et al.,* (2015) researched on the tribological activity of cotton seed oil as a friction and wear controller for application in multi-cylinder engine by evaluating the anti-wear characteristics of cottonseed oil. The study used four ball testing machine based on American Society of Testing and Materials (ASTM) ASTM D 4172 specification (ASTM, 2010) to quantify the friction coefficient and diameter of wear wound of cotton seed oil and compared it with commercial mineral oil lubricants SAE 20W50 and SAE 20W40.The test Balls were manufactured using chrome alloy steel from AISI standard steel E-52100, having diameter of 12.7 mm (0.5 in.) and Grade 25 EP (Extra Polish), and has 64 to 66 Rockwell hardness C. It was found that though coefficient of friction for cottonseed oil was 0.0636 which is compared to the commercial lubricants SAE 20W50 with friction coefficient of 0.1121 and SAE 20W40 with friction coefficient of 0.086 was lower, the wear wound diameter for cotton seed oil exceeded that of the commercial mineral oils as seen in Table 2.2. Hence it was concluded that cotton seed oil cannot be used in the unmodified form because, though coefficient of friction for cottonseed oil is small compared to commercial lubricants, the wear wound diameter is larger, the wear characteristic might be made better via chemical modification or suitable anti-wear additives addition.

Table 2.2 Mean values of friction coefficient and wear wound diameter for tested oils

Lubricant/Oil	Coefficient of friction (μ)	Wear scar diameter (micron)
SAE 20W50	0.1121	523.55
SAE 20W40	0.086	401.44
Cotton seed oil	0.0636	653.44

(Source: Kalhapure, *et al.,* 2015)

2.2.3 Sesame oil as potential crop base stock for eco-friendly industrial lubricants

Nair *et al.* (2017), carried out detailed study on the evaluation of physicochemical, thermal and tribological properties of sesame oil (*Sesamum indicum L.*) as a potential agricultural crop base stock for eco-friendly industrial lubricants. The physicochemical, rheological, thermal, oxidative, and tribological properties of sesame oil were studied and compared with coconut oil, sunflower oil and a commercially available mineral oil, SAE 20W40 using standard ASTM methods.

The sesame oil was extracted from sesame seeds collected from Hooghly, East Midnapore and West Midnapore districts of West Bengal, India. The fatty acid foot print of plant seed oils were captured using Gas Chromatography, the stability in the presence of heat was evaluated using thermogravimetric analysis (TGA). The equipment used to evaluate TGA had a capacity of 0–400°C. To determine the pour point, Mettler Toledo Differential Scanning Calorimetry (DSC) 822e was used; the testing was done between –50°C to 80°C and sample weight ranging from 6 mg to 9 mg.

The conventional ASTM D97-96a method (ASTM, 2002a) was also performed to evaluate pour points of the selected oils and to verify the results obtained from DSC. Rheological properties of the sesame oil, coconut oil, sun flower oil and the petroleum oil SAE 20W40 were determined by evaluating their viscosities. Kinematic viscosities at 100°C and 40°C were estimated using Redwood viscometer, from these results viscosity index (VI) for the oils were calculated per ASTM D 2270 (ASTM, 1998a).

The friction-wear characteristics of the oils were evaluated by measuring the diameter of wear wound (WSD) and coefficient of friction (CoF), using a four-ball tester according to the ASTM D 5183-05 standard for coefficient of friction (ASTM, 2016). The load, rpm, run-time and the temperature for the COF test are 40 kg, 600 rpm, 60 minutes and 75°C respectively. ASTM D 4172-94 (ASTM, 2010) standard was used to conduct wear test. The load, rpm and run-time and the temperature for the wear test are 40 kg, 1200 rpm, 60 minutes and 75°C respectively. The WSD was analysed using an illuminated microscope and a Hitachi SU6600 Electron Scanning Microscope (SEM). The balls used for the tests were of diameter 12.7 mm and made from chrome alloy steel with a Rockwell hardness of 61 HRC.

It was found that from the flash and fire point data that the sesame oil was more thermally stable than commercially available mineral lubricant SAE 20W40. Also pour point of the sesame oil was better than that of the coconut oil; which was attributed to the coconut oil having a higher level of saturated fatty acids than the sesame oil (Jayadas & Nair, 2006). Thus, raw sesame oil (without using any pour point depressant additives) has pour point comparable to commercially available mineral oil SAE 20W40. The result of the thermal properties is shown in Table 2.3 and the tribological and rheological properties for the vegetable oils studied are shown in Table 2.4. All vegetable oils had high VI since the change in viscosities with temperature was less. Among the selected vegetable oils, sesame oil possessed the highest viscosity index. However, its viscosity index was much lower than that of the mineral oil SAE 20W40. Therefore, sesame oil can be employed as a commercial lubricant if its viscosity index is improved further by the addition of proper viscosity improvers.

Table 2.3 Thermal properties of Some Selected oils

Oils/lubricant	Thermal Properties(°C)		
	Flash Point	Fire Point	Pour Point
Sesame	315	319	-15
Coconut	320	325	22
Sunflower	332	337	-18
SAE 20W40	204	209	-20

(Source: Nair *et al;* 2017)

Table 2.4: Rheological and tribological properties of Some Selected oils

Oils/lubricant	Viscosity at 40°C (cst)	Viscosity at 100°C (cst)	Viscosity Index	Coefficient of friction	Wear scar diameter (mm)
Sesame	31.86	7.46	213.5	0.0862	0.650
Coconut	27.8	6.1	176	0.0901	0.836
Sunflower	29	6	159	0.0742	0.685
SAE 20W40	112.6	14.8	135.57	0.107	0.470

(Source: Nair *et al*., 2017)

The tribological studies showed that the coefficient of friction of the sesame oil was much lower than that of coconut oil and mineral oil SAE 20W40. However, the wear scar diameter obtained for SAE 20W40 was better than that of vegetable oils. Among the vegetable oils, wear scar diameter obtained for the sesame oil was less than that of coconut oil and sunflower oils. By adding proper anti-wear additives to sesame oil, wear can further be brought down. It was concluded that, in general, sesame has excellent thermal and tribological properties, but the improvement of its oxidative and rheological properties are essential. Sesame oil can be considered as a potential

agricultural crop base stock for industrial lubricants which can become an eco-friendly substitute for its mineral oil counterparts in near future.

2.2.4 Date pulp as potential environmentally friendly lubricant

Mohammed (2017), preliminarily evaluated the tribological properties of date palm fruit syrup as a potential environmental-friendly lubricant using a ball on disc tribometer. Different amounts (50, 75, and 100 vol %) of the pulp in water were evaluated at anormal load of 5 kg and a sliding straight-line velocity of 0.1 m/s. electron optical profilers were used to characterize the wear tracks and estimate the wear rate.

The totally date pulp without water with a viscosity of 0.01695 Pa showed impressive outcome by subduing the friction coefficient of steel disc and ball pair from 0.6 (dry conditions) to a value of ~0.1. The depth of the wear track reduced from ~152 μm (dry conditions) to ~11 μm, signifying a considerable reduction in wear. The coefficient of friction of 100 % date palm syrup was at par with a commercial lubricant, also the wear track depth was slightly less for 100% syrup as compared to industrial lubricant. The width of the wear track was less for the 100% syrup (~316 μm) when compared to the industrial lubricant (~328 μm). However, the thermo-oxidative as well as temperature properties of the syrup were not studied and are likely not to be good.

2.2.5 Blends of various vegetable oil as lubricant

Talkit *et al*., (2015), carried out an empirical study on lubricating characteristics of different plant oils mixed at different temperatures. Testing of soybean oil, olive oil, almond oil, amla oil, castor oil, groundnut oil, cottonseed oil, coconut oil, sesame oil, sunflower oil and mustard oil and their blends in different proportion for friction, wear

viscosity, cold flow high temperature characteristics and percentage carbon chaff was carried out.

It was found that soybean almond oil blend possessed the lowest pour point which was attributed to increasing cis-unsaturation or low molecular weight fatty acid while soybean amla oil blend possessed the highest pour point due to increasing high molecular weight fatty acid. Also, soyabean oil combined with castor oil possessed the least cloud point while combination of soybean sesame oil had most cloud point. Soybean groundnut oil blend had highest flash point while soybean castor oil blend had the lowest flash point. Similarly, it was found that soybean olive oil blend had the highest fire point while soybean almond oil blend possessed the lowest fire point; the percentage carbon chaff of soybean amla oil blend was lower while soybean castor oil blend had higher carbon remains than other plant oil blends. The study is useful for development of biolubricants from blends of soya bean and other vegetable oil however it is deficient because tribological evaluation of the oils was not carried out.

Similarly, Mannekote and Kalas (2012), evaluated the effect of oxidation on the tribological performance of few vegetable oils. Ground nut oil, soyabean oil, rice bran oil, sesame seed oil and palm oils were allowed to undergo oxidation and after wards the oxidaised oils were subjected to tribological tests using four ball tribometer. The result showed that oxidation of the vegetable oils negatively affected its tribological properties. However, the use of these edible oils as lubricants will not be welcomed by the public as there is scarcity of edible oils for food purposes.

2.2.6 Effect of nano particles on tribological performance of vegetable oil lubricant

Alves *et al*. (2013), investigated the tribological behaviour of vegetable oil based lubricants with nano particles of oxides in boundary lubrication conditions. Sunflower and soybean oils were epoxidized and analysed in terms of viscosity, density and iodine value. Copper oxide and zinc oxide nano particles were prepared by an alco-thermal method and added to the vegetable oils, mineral oil and synthetic oil that were used as control. Steel disc-ball machine was used to carry out the wear-friction tests. Results depicted that friction and wear characteristics of the petroleum-based oils appreciated significantly by the introduction of the nano particles of zinc and copper oxides, however, nano particles addition did not better the tribo-characteristics of vegetable oils. Thus, copper oxide and zinc oxide nano particles do not depict acceptable anti wear ability when mixed with epoxidized vegetable oils.

Additionally, Shafi *et al.* (2018), reviewed the use of nanoparticles in some edible vegetable oils as lubricants. The friction and wear performance of the vegetable oils containing nano particles were evaluated using standard tribometers. The results showed that the nanoparticles gave little improvement on the tribological performance of the edible vegetable oils. The reviewed work did not consider the effect of the nano particles on the biodegradability of the vegetable oils and the use of edible vegetable oils as lubricant is not acceptable by the public due to scarcity of edible oils for food purposes.

2.2.7 Vegetable oil- petroleum oil blend based lubricants

Vegetable oil has better lubricity compared to mineral oil base lubricants therefore it is added to mineral oil as lubricity additive. Bahari *et al.* (2017) studied the friction coefficient and wear rate of palm and soybean blends with commercial petroleum based engine oil SAE 15W40 for high temperature and severe contact conditions application using a linear reciprocating rig. Four samples were prepared: palm oil,

soybean oil, 50% mineral oil+50% palm oil and 50% mineral oil and tested for friction, wear, viscosity, and oxidation stability.

Results obtained showed that mineral oil had the lowest friction coefficient (0.093) while the soybean oil had the highest (0.112). Also, the wear protection of the petroleum oil was the best as only 0.65 mg mass loss was observed; palm oil had the worst wear performance of 45.76 mg reduction in mass. The blends of oil palm with mineral-based oil showed 25% reduction in wear compared to raw oil palm while soybean oil with mineral oil blend showed 27% improvement. It was concluded that there is no simple relationship between wear and friction, the palm and soybean oils performed below the commercial mineral oil and are therefore not suitable as severe contact engine oil or lubricity additive.

2.2.8 Improvement in oxidative stability of vegetable oil based lubricants

Erhan *et al.* (2006), studied the resistance to oxygen attacks and cold flow characteristics of vegetable oil based lubricants by adding Zinc diakyl dithiocarbamate (ZDDC) and Antimony diakyl dithiocarbamate (ADDC) to five vegetable oils (refined soya bean oil, mild oleic soya bean oil, high oleic soyabean oil, highly enriched safflower and h sunflower oils) then evaluating their pour point as per ASTM D97 method (ASTM, 2002a), oxidation and low temperature stability using differential scanning calorimetry (DSC) and rotating bomb oxidation tests (RBOT) as per ASTM D-2272 (ASTM, 2002b) standard and comparing the result to that of a commercial biodegradable hydraulic fluid. The results showed that the vegetable oils formulated according to the above methods exhibited higher oxidation resistance and better low temperature characteristics compared to the commercial hydraulic fluid and compared at the same level with mineral based lubricants.

2.2.9 Jatropha oil in mineral oil lubricants

Shahabuddin *et al*. (2013a), studied the creation of environmentally compatible biodegradable oils made from jatropha oil mixed in mineral based oil (SAE40) using a 3-pin-upon-disc machine. Aluminium was employed to mould three pin and cast iron was used for disc sample while lubricant specimens gotten from homogeneous stiring of 10%, 20%, 30%, 40% and 50% Jatropha oil in SAE 40 was obtained. Friction, wear and Viscosity were tested for both 40°C and 100°C. Also, a many-element oil analyser (MOA) was used to ascertain the types and quantity of metals contained in the lubricating oil at the end of the experiments.

The results showed that the rate of wear for 10% Jatropha oil introduced to the lubricant was almost the same with parent lubricant material. From the chemical constituent analysis of the bio lubricants, it was found that amount of Iron and Aluminium appreciated after the test due to the wear of the material from the pin and the disc members. In terms of rheology, about all plant based lubricants fulfilled the requirement for ISO viscosity standard whereas, 40% and 50 % mixture of Jatropha oil fall short of the ISO VG 100 requirement at 40°C. Though impressive results were obtained, yet the biodegradability and environmental friendliness of the lubricant developed cannot be ascertain as the use of hazardous mineral oil persists.

In furtherance to the research, Shahabuddin *et al*. (2013b) also carried out a comparative tribo-analysis of plant based lubricant made out of non-food oil source (Jatropha oil) with the aid of four-ball tribo test and cygnus wear test machines. To blend the plant based lubricants, 10–50% by volume of jatropha oil were introduced into the base oil SAE40. Empirical results depict that boundary layer lubrication was the dominant regime during the test, the major wear processes were abrasive and adhesive wear. In the process of wear testing, the smallest wear was discovered during

the introduction of 10% jatropha oil, beyond 20% presence of impurity, the rate of wear was muchly enlarged. The introduction of Jatropha oil in the parent oil acted as a desirable lubricant enhancer which brought down the friction coefficient and diameter of wear by 34%. The introduction of 10% Jatropha oil in the parent oil produced the best result for automobile engine purposes as it gave the best overall result considering wear, coefficient of friction, viscosity, temperature rise, wear scar diameter and flash temperature parameter (FTP).

Also, the Effect of bio-lubricant on tribo-characteristics of IP 239 standard steel was investigated by contaminating SAE 40 with various amounts (1%, 2%, 3%, 4%, 5% by volume) of jatropha oil. The friction torque and wear were studied using four ball tribotester. It was found that diameter of wear moved up proportionally as the load addition for lubricant oil and came down again by addition of percentage of jatropha oil. Frictional forces analysed in the investigation showed that 5% of jatropha oil introduced had dominating characteristics at 40 kg load. The 1 out of 20 parts of jatropha oil introduced into the base oil gave better out come in terms of tribological properties and was suggested to be alternative lubricant for the automobile engine applications especially for steel (Habibullah *et al.,* 2014). This study did not consider the thermal oxidative stability of the formulated lubricant and the environmentally unfriendly mineral oil is still the base oil.

2.2.10 Comparison of jatropha and palm oil lubricants tribological properties

Heikal *et al.* (2016), studied the manufacturing of environment friendly biolubricants from palm and jatropha oils. The vegetable based lubricants were produced through a two stage transestrification, confirmed by Fourier Transform Infrared (FTIR) spectroscopy and analysed for viscosity, pour point and thermal stability according to ASTM D4172 and ASTM D2270 standard methods (ASTM, 2010). It was found that

the jatropha oil based biolubricant exhibited high viscosity index (140), desirable small pour point (-3^{0}C) and manageable stability at high temperature and thereby meet the requirement for commercial industrial oil ISO VG46 grade.

The palm oil based biolubricant had high pour point (5^{0}C) which is undesirable and therefore needs improvement. However, its viscosity, viscosity index and flash point are at par with commercial industrial oil ISO VG32 and VG46. The friction and wear properties of the developed biolubricants where however not consider.

2.2.11 Castor oil lubricants

Castor oil is gotten from the castor bean tree (Ricinus communis). It is a member of the family Euphorbiaceae. It is a drought resistant non-edible oil seed crop widely found in every hot and mildly hot climate, but also in many of the hot countries of the globe. Oil from castor is considered a significantly important laxatives, its annual Indian yield is 271 MT. Its seed contain 46 to 55% oil that is viscous, non-volatile, non- drying, pale yellow in colour with slight odour and a bland taste.

Binfa *et al.* (2015) carried out a study to compare the lubricant properties of castor oil and commercial engine oil (20/W50) using a steel ball (comparable to EN 31, 64–66 HRC type), 12.7mm in diameter four ball tribo-tester. The test for friction coefficient and wear scar diameter was carried out based on ASTM D4172 method (ASTM, 2010), while the extreme pressure test was based on IP 239 method, similar to ASTM D 2783 (ASTM, 2003). The result showed that castor oil a lower coefficient of friction of 0.0607 compared to the commercial mineral oil with a coefficient of friction of 0.0763, however the wear scar diameter of the commercial mineral (0.40322) was lower than that of the castor oil (0.68625). Also, in the extreme pressure test, Castor oil demonstrated a higher weld load (2158N) compare to engine oil (1766N). It was

concluded that Castor oil can reduce friction and improve fuel conservation and mechanical efficiency in engines more than the commercial mineral-based oil because unrefined castor oil possesses better "oiliness" and higher weld load, than formulated foreign commercial 20W-50 crankcase oil.

2.2.12 Improvement of vegetable oil based lubricants properties

Quinchia *et al*. (2013) researched tribological potential of plant oil-based lubricants containing eco-friendly viscosity modifiers. Enriched oleic sunflower oil, soybean and castor oils were blended with 4% (w/ w) of ethylene–vinyl acetate copolymer (EVA) and 1% (w/w) of ethyl cellulose (EC). The viscosity over a temperature range of 25 – 100 ^{0}C were measured as well as the thin layer thickness and opposition to motion measurements were done. It was discovered that oil from castor bean gave the most desirable behaviour in comparison to the sun flower and soya bean oils. The EC was more competitive than EVA as a viscosity adjusting material with the best viscosity increments (125% at 40^{0}C) corresponding to castor oil- ethyl cellulose blend.

Azhari *et al*. (2015) studied the modification of corn and canola oils using ZDDP as wear additive. Specimens of oils were prepared without addition of ZDDP, with addition of 2 wt% and 5 wt% ZDDP. The formulated were tested using rotary disc electrode atomic emission spectroscopy (RDE-AES) which is monitored by spectroil (computer) software, and a heated viscosity measuring instrument. The specimen analysis was then done using a pin-on-disc tribo-tester. Corn oil had smaller kinematic rheology when placed side by side with canola oil and produced a lower kinematic rheology value of 36.3 cSt with 2 wt% ZDDP addition. The inoculation of 2 wt% ZDDP in the oils lowered the friction coefficient for both oils. It is observed that 5% addition of ZDDP will not lead to further improvement of lubricant properties in corn and canola oils.

Karmakar *et al.* (2017), reviewed the chemistry of vegetable oils modified using different chemical processes. The vegetable oils reviewed were subjected to different processes of chemical modification with the aim of developing green lubricants. The chemistry of the modified vegetable oils was later studied. The results showed that esterification, transesterification, hydrolysis and thermal cracking succeeded in altering the chemical bonds of the vegetable oils and made them more suitable for application as lubricants. Attention was not given to the tribological performance of the modified vegetable oils.

Similarly, Onuh *et al*. (2017) investigated the application of transesterified jatropha oil as a lubricant in water-based drilling mud. The result indicated that drilling mud formulated with transesterified jatropha oil had desirable properties for drilling applications. However, the tribological evaluation and biodegradability of the lubricant was not considered.

2.2.13 Taguchi method in lubricant research

Singh (2015) reviewed the importance of deploying Taguchi method reported by many investigators. These literatures review the use of lubricants and design of experiments. Study of Silica reinforced composites under various measurement conditions which include solid lubricants, sliding velocity and force was done. Solid lubricants were h-BN graphite and MoS_2, forces of 3000 to 9000 kg, and sliding velocity between 3.0 to 9.0 m/s were used. Under these conditions, MoS_2 distinguished itself as an effective wear resistance and both h-BN and MoS_2 provide better braking performance at higher sliding speed and force (Prabhu, 2015). Design of experimental procedure was used to select best production-options of octyl esters from fatty acid that are mobile towards adding more potential to plant based lubricant. Catalysed organic neutralisation reaction of waste cooking oil and octanol was used. The input factors included moles

of octanol, temperature and duration of reaction. It was discovered that the product exhibited greater flame temperature, index of rheology and 9 out of 10 parts biodegradability over the classical waste cooking oil used (Chowdhury *et al.*, 2014). Kapsiz *et al.*, (2011) also documented that cylinder liner/piston ring reciprocating tests based on Taguchi method were investigated under various testing situations to optimize minimum piston ring weight loss and friction. Sliding velocity, lubricant and force were input parameters. The interaction between sliding velocity and oil type dominated least weight loss development model

It can be summarised using available literature that scanty research documentation exists on vegetable oil -based lubricants. One of the objectives of this study is to optimize some tribology-controlling inputs like concentration of anti-wear additive, concentration of viscosity modifier, concentration of anticorrosion additive and extreme pressure additive in lubricants using design of experiment via Taguchi method to analyse dominant controlling parameters on the friction coefficient, viscosity index, and rate of wear.

2.3 Research Gap

The reduction in global petroleum reserve, fluctuating petroleum price as well as several issues related to environmental degradation necessitated renewed interest in application of plant-based lubes. Vegetable oil based lubricants are emerging development considering the advantages and disadvantages of vegetable oils. In the absence of additives, plant oils out-performed petroleum based oils although it manifests poor low temperature character, bad stability in the presence of heat and oxygen; vegetable oil also exhibit lower effectiveness under extreme loads. Even though previous studies have established the superiority of mineral oil over vegetable

oils for extreme loads operations, there should be some way to overcome this problem so plant oils can be used for stamping or hydraulic applications. Table 2.5 is a summary of some of the newest research on jatropha and castor oil based lubricants and the identified research gap. From the Table it is deduced that research on the wear and friction performance of lubricants developed from jatropha and castor oils is an area needing attention and this research seeks to address this gap.

From the reviewed literature, previous research concentrated on unmodified vegetable oil or addition of vegetable oil to mineral oil and this come with all the attendant limitations as earlier mentioned. Limited work has been done about modification of vegetable oil for industrial and internal combustion engines lubricants applications. Additionally, scanty literature exists on the use of additives in the formulation of vegetable oil based lubricants. In few instances where additives are applied, heavy metal (zinc and antimony), sulphur and phosphorous containing additives which are hazardous are included in the additives. There is therefore a need to look into the use of environmentally friendly additives and the most significant additives in the formulation of vegetable oil based lubricants, which is one of the objectives of this work.

Table 2.5: Summary of researches on vegetable oil- based lubricants and the identified gap

S/N	Reference	Lubricant Investigated	Contribution	Identified Gap
1	Maleque *et al.,* (2003)	Palm oil	Developed lubricant additive from palm oil methyl ester	Food versus lubricant debate challenge is not yet solved
2	Shashidhara *et al.,* (2012)	Pongam and jatropha oils	Developed lubricants from raw and modified non edible vegetable oils	The effect of additives on the developed lubricants was not studied and the oils performed poorly in wear prevention
3	Imran *et al.,* (2013)	Mineral oil and jatropha oil	Evaluated the tribological properties of blended jatropha and mineral oil lubricant	The biodegradability of the blend was not studied and the use of mineral oil and its problems still persist
4	Garba *et al.,* (2013)	Jatropha Curcas Seed oil	Degummed jatropha oil meets the ASTM standard for use as transformer oil.	The corrosion inhibition properties and biodegradability of the oil was not studied.
5	Bilal *et al.,* (2013)	Jatropha Curcas Seed Oil	Transesterification of Nigerian Jatropha oil with ethylene glycol improves pour point and gives a lubricant conformable to ISO VG-46	Iodine and peroxide values, cloud and flash points and biodegradability of the Nigerian Jatropha oil were not studied.
6	Mohammed, (2015)	Jatropha, Moringa seed, castor and cotton seed oils	Transesterification of Nigeria Jatropha, Moringa seed, castor and cotton seed oils with trimethylolpropane improves pour point but degrades rheological properties.	Iodine and peroxide values, cloud and flash points and biodegradability of the Nigerian Jatropha oil were not studied.
7	Binfa *et al.,* (2015)	castor oil and commercial engine oil	Pure oil from castor bean carry better "lubricity" and welding load, than foreign 20W-50 crankcase oil found in market	The wear performance of the castor oil needs to be improved so that it can compete with SAE 20W-50 engine lubricant

8	Zulkifli *et al.,* (2015)	Palm oil	Developed biolubricant from palm oil and polyols	Use of palm oil will give rise to food versus lubricant debate and physicochemical properties of the biolubricant was not measured
9	Farhanah *et al.,* (2015)	Mineral oil based engine oils	Demonstrated that different engine oils in the market having the same SAE viscosity grade will not always have the same lubricity	Mineral oil based engine oils are not eco-friendly
10	Menkiti *et al.,* (2015)	Fluted pumpkin seed	Optimised synthesis of biolubricant from fluted pumpkin seed	Tribological evaluation and the use of additives was not carried out
11	Musa *et al.,* (2016)	Jatropha Curcas Oil	Optimised the process conditions for production of biolubricant from Nigeria jatropha oil and Trimethylolpropane.	Focused on the yield of biolubricant from the process without paying attention to the properties of the jatropha oil or the produced biolubricant.
12	Mamuda, *et al.,* (2016a)	Neem seed oil and jatropha curcas seed oil	Nigeria jatropha oil formulated with Antimony diakyldithiocarbamate (ADTC) is suitable for production of wire from mild and medium iron-carbon alloy rods	Physicochemical, temperature, rheological, corrosion inhibition properties and biodegradability of the Nigerian jatropha oil were not studied.
13	Mamuda, *et al.,* (2016b)	Jatropha.Curcas Seed Oil and 10W-30	Jatropha oil performed better than 10W-30 in friction reduction but less in wear protection.	Physicochemical, temperature, rheological, corrosion inhibition properties and biodegradability of the Nigerian jatropha oil were not studied.
14	Atuci *et al.,* (2016)	Low price bulk mineral oil based engine oils	Investigated commercial mineral oils in Nigerian markets	Mineral oil based engine oils are harmful to man and the environment
15	Bhaumik and Pathak (2016)	Commercial Mineral Oil and Neat Castor Oil	Castor oil performed less than 372 cSt heavy duty mineral oil lubricant in friction, wear, extreme pressure, viscosity and scuffing.	Evaluation and modification of the castor oil needs to be carried out for it to compete with commercial heavy-duty lubricant.
16	Kania *et al.,* (2017)	Palm oil	Developed drilling lubricant from palm oil and polyol esters	Use of palm oil will give rise to food versus lubricant debate and biodegradability of the biolubricant was not measured

It has been concluded from the available literature reviewed that very limited work and scanty literatures exist on the study of the effect of additive concentration on the tribological behaviour of biolubricants. There is little or no literature on then most significant of Zinc diakyldithiocarbamate (ZDDC), Triacyl triphosphate (TCP), Cetyl chloride and hydroxethyl cellulose (HEC) on the tribological performance of lubricants. This work will evaluate the tribological performance of lubricants developed from castor oil and jatropha oil blended with these four selected additives.

Many researchers in the past worked on edible vegetable oil as base stock in lubricant formulation. Presently there is increasing concern about the use of edible vegetable oil for lubricant formulation. This work will use non-food plant oils (jatropha and castor oils) that are locally available to develop lubricants for industrial applications.

CHAPTER THREE

3.0 MATERIALS AND METHODS

3.1. Materials

The materials used in the research are Jatropha oil and castor oil sourced from Agrienergy Kano. Also, a mineral oil based, multigrade commercially available lubricant (SAE 20/W50) obtained from Ammasco International Limited Kano was also used for comparison purpose. Distilled water and Potassium methoxide were prepared in the Water Aquaculture and Fisheries (WAFT) laboratory, Federal University of Technology (FUT) Minna. In addition, chemical reagents and lubricant additives were used. The detail description and discussion of the other materials used is carried out in the following subsections.

3.1.1 Chemicals

The list of chemicals used for this research is as shown in Table 3.1.

Table3.1: List of Chemicals Used

S/N	Chemical	Content Purity	Distributor
1	Methyl alcohol	99.5 %	JHD Chemical Reagents Co. Ltd, Guangzhou
2	Trimethyloloropene (TMP)	98.0 %	Lobachemie Private Limited
3	Potassium hydroxide	98.0%	Qualikems
4	Distilled water	99.9 %	WAFT Lab
5	Sulphuric acid	99.0%	JHD Chemical Reagents Co. Ltd, Guangzhou
6	Isopropyl alcohol	99.0 %	JHD Chemical Reagents Co. Ltd, Guangzhou
7	Phenolphthalein indicator	Analar	JHD Chemical Reagents Co. Ltd, Guangzhou
8	Potassium methoxide (30% in methanol)	99.0 %	WAFT Lab

3.1.2 Laboratory equipment

The laboratory equipment/instruments and source are outlined in Table 3.2.

Table 3.2: List of laboratory instruments/equipment used

S/N	Instrument/Equipment	Source
1	Digital weighing balance	W.A.F.T Laboratory
2	Stirrer (magnetic)	Agric. Engrg. Laboratory
3	Evaporator (rotary)	Biochemistry Laboratory
4	Stand (retort)	W.A.F.T Laboratory
5	Conical flask	W.A.F.T Laboratory
6	Pipette	W.A.F.T Laboratory
7	Oven	W.A.F.T Laboratory
8	Separating funnel	W.A.F.T Laboratory
9	Beakers	W.A.F.T Laboratory
10	Three necked bottom flask	Agric. Engrg. Laboratory
11	Leibig condenser	Biochemistry Laboratory
12	Vacuum pump	Biochemistry Laboratory
13	Thermometer (500°C)	Agric. Engrg. Laboratory
14	Water bath And stirrer	WAFT Laboratory
15	GC analyser	ABU Zaria
16	FTIR analyser	ABU Zaria
17	DTA/TGA analyser	Step B FUT Minna
18	UV Spectrometer	Microbiology Laboratory
19	Digital pH meter	W.A.F.T Laboratory
20	Viscometer	Agric. Engrg. Laboratory

3.1.3 Lubricant additive

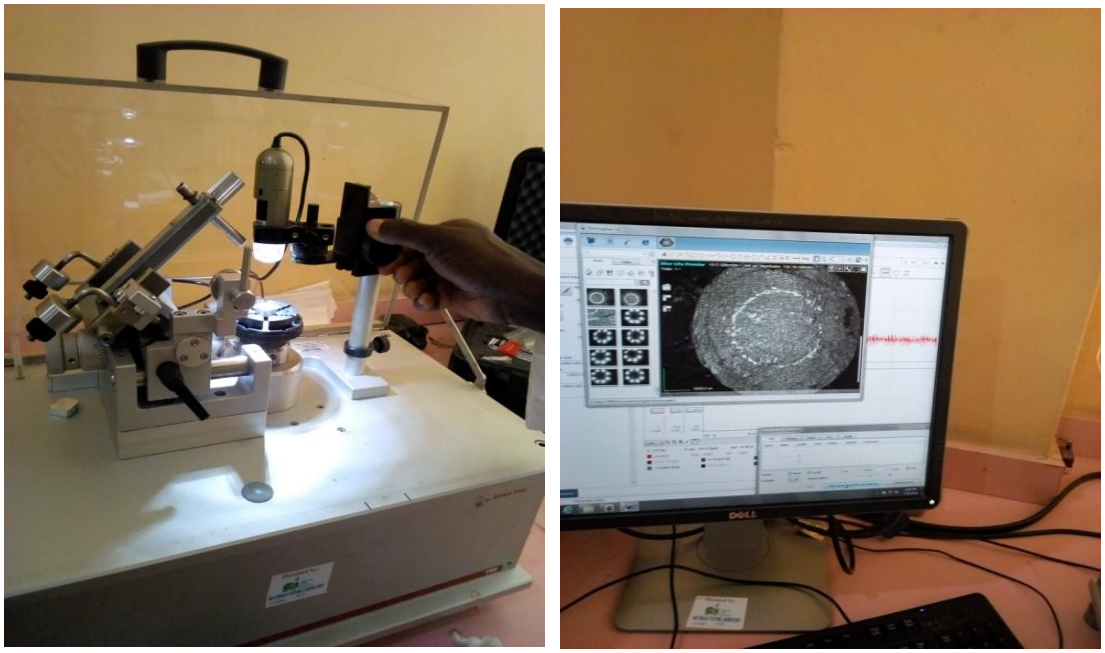
The details of the four additives used in this work are listed in Table 3.3.

Table 3.3: Additives deployed for the research

S/N	Additive	Composition	Symbol
1	Anti-wear	Tricrysl Phosphate (TCP)	A
2	Viscosity modifier	Hydroxy ethyl cellulose (HEC)	B
3	Extreme pressure	Cetyl chloride	C
4	Anti-corrosion	Zinc diakyldithio carbamate (ZnDDC)	D

3.1.4 Tribometer

The Anton Paar standard tribometer TRN version 6.1.19 shown in plate I was used for the tribological evaluation of the oils and lubricants. The TRN model standard tribometer is an Austrian design which can be used as a pin-on disc or ball-on-disc tribometer. It has an electronic microscope fitted for wear measurements and has several sensors connected to a computer for data acquisition and processing.



(a) (b)

Plate I: The Anton Paar standard Tribometer TRN model (a) tribometer (b) computer monitor screen showing image of disc being measured for wear

3.2 Methods

3.2.1 Characterisation and modification of jatropha and castor oils

The jatropha and castor oils in this work were first standardized by characterizing their properties. These properties include; physicochemical, rheological, temperature, thermo-oxidative stability and corrosion prevention properties as well as the biodegradability of the oils. The jatropha and castor oils were also chemically modified to improve their properties and improve their suitability for industrial lubricant applications. Details of the characterisation and modification of the oils is given in the following subsections.

3.2.1.1 Vegetable oils chemical and physical characteristics measurement

Chemical and physical properties (density over water density, acid level, percentage free acid of fat origin, iodine level and pH) of the jatropha and castor oils were analysed in the Water

Resources and Fisheries Technology Laboratory, FUT Minna. Details of the analysis are reported in the following subsections.

i. Determination of specific gravity and density

The density was measured according to the ASTM D1298 standard while the specific gravity was determined according to the ASTM D1217 standard (ASTM, 1998b). A 50 ml SEDI-M bottle for density value was cleaned very well using liquid soap and water followed by pet-ether. After which it was oven dried followed by measuring its mass. The bottle was filled with distilled water and weighed, dried again and filled up with the oil specimen and weighed as shown in Plate II. From theory, the specific gravity is the mass of the oil weighed divided by the mass of water weighed and the density of the oil was the ratio of mass to volume of the oil.

(a) (b)

Plate II: Determination of specific gravity and density (a) oil in the density bottle (b) weighing of oil and density bottle

ii. Determination of pH of the vegetable oils

The pH value of any fluid indicates the general condition of acidity or alkalinity of the fluid. Lubricants with very low or high pH value can be hazardous to human operators; also, microbial contamination of lubricants takes place in acidic medium rather than alkaline medium. It is also known that an acidic (low pH value) will reduce the corrosion protection of the machine parts being lubricated.

The pH values of the seed oils were measured with the aid of the REX pHS -25 model Ph meter shown in plate III. The pH meter was first calibrated using a solution of known pH. The electrode of the pH instrument was cleaned with distilled water before taking another reading.

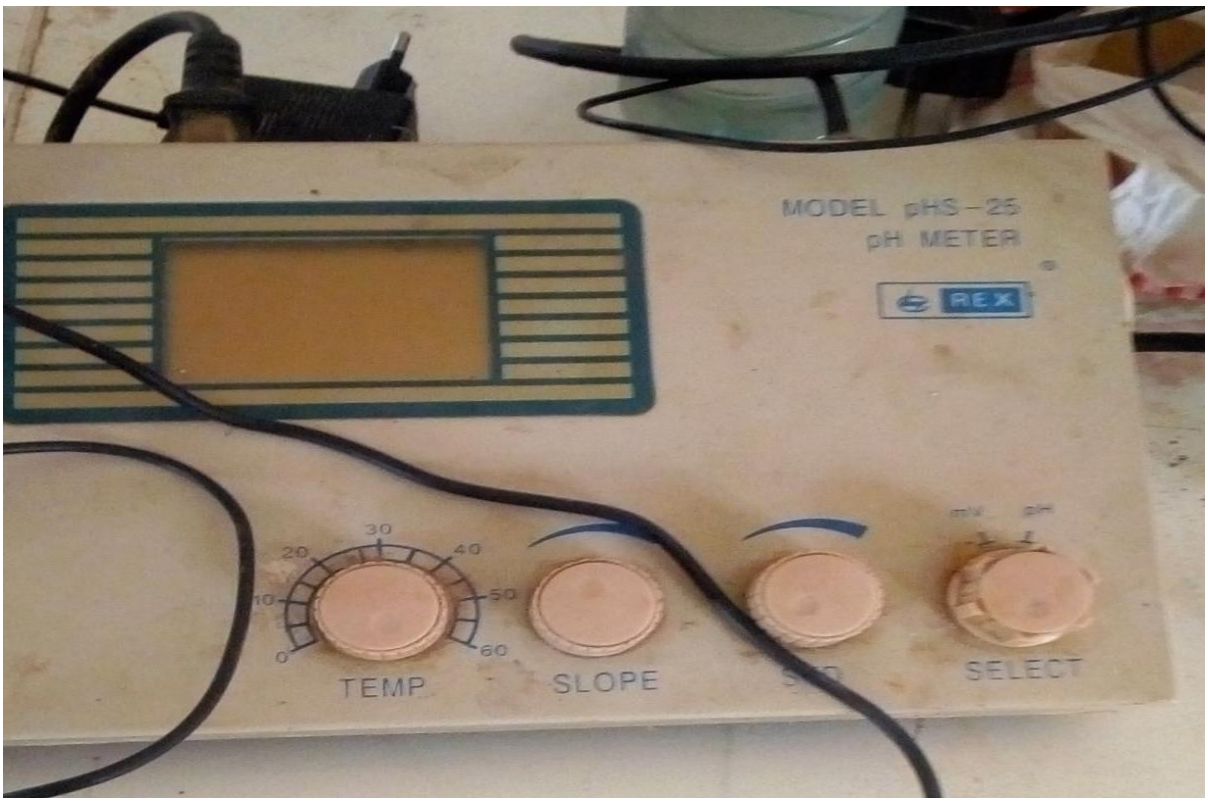


Plate III: REX pH meter

iii. Vegetable oils saponification value quantification

The alcoholic KOH was freshly prepared by dissolving KOH pellet in ethanol. More than 1g of oil was measured and poured into a conical flask, then 25 ml of the alcoholic KOH was

added to it, a blank was also used. The sample was well covered and placed in an oven for 30 minutes shaking it periodically, 1 ml of phenolphthalein was added to the mixture and to the blank and titrated against 0.5M HCl to get the end point. The saponification value (SV) was calculated using equation (3.1)

$$\mathrm{SV} = \frac{56.1\mathrm{X}(\mathrm{B}-\mathrm{A})\mathrm{XN}}{\mathrm{Woil}} \qquad (3.1)$$

Where; B= volume of standard ethanol potassium hydroxide used in blank titration; A= volume of standard ethanol potassium hydroxide used in titration with the oil; N= normality of standard acid; and W_{oil} = weight of oil used.

iv. Determination of saturation level of the vegetable oils

The oil was transferred into a small beaker; a small rod was added to it. About 0.001 to 0.002 kg of the oil was taken and introduced into a 250 ml capacity glass-stopper bottle. Then 0.010 litres of carbon tetrachloride was mixed with the oil to dissolve. 0.020 litres of Wiji's solution was dropped in and a cork was used to cover it and left to it was left in the dark for 0.5 hours. Then 1.5 cm^3 of potassium iodide water (1 out of 10 parts) and 0.1 litres of water was brought in and the mixture was vigorously shaken and neutralised with 0.1 M sodium thiosulphate solution with starch-indicator (titration = 'A'ml). A blank was also carried out at the same time starting with 10 ml of carbon tetrachloride (titration = 'B'ml). The iodine value (IV) was calculated using the equation (3.2)

$$\mathrm{IV} = \frac{0.1269X(B-A)XNX100}{Woil} \qquad (3.2)$$

Such that B = volume of sodium thiosulphate used in blank titration. A= volume of sodium thiosulpate used in titration with oil. N= normality of sodium thiosulphate. W_{oil}= weight of oil used and 0.1269 is the iodine number.

v. Quantification of acid level and percentage acid from free fat (%FFA)

The acid content of oils investigated was determined by taking 0.002 kg of the oil that was poured into a 250 ml glass bottle. A non-reacting mixture of pet-ether and alkanol) was prepared and 50 ml was transferred into the glassware containing the oil sample. The mixture was manually stirred vigorously for ½ Hrs. 0.56 g of potassium hydroxide (KOH) powder was taken and transferred into a separate beaker and 0.1M KOH was made, 3 drops of phenolphthalein indicator was added to the oil-ethanol-petroleum ether mixture and was titrated against 0.1M soap and observed for colour to have turned pink and persisted for 15minutes. The acid value (AV) was determined using the equation (3.3).

$$\text{AV} = \frac{56.1XVXN}{Woil} \tag{3.3}$$

Such that; V= volume of standard alkali used; N= normality of specified alkali used;

W_{oil}= used oil weight.

The percentage free fatty acid (%FFA) is gotten from equation (3.4)

$$\text{FFA} = \frac{AV}{2} \tag{3.4}$$

where FFA = free fatty acid and AV = acid value.

3.2.1.2 Determination of rheological and temperature properties

Rheology is the science of the flow of matter. The most important rheological parameter for lubricants is viscosity. Tribological characteristics such as interacting surfaces friction coefficient and wear depends on the viscosity of the lubricant used. Viscosity index and viscosity-pressure relationship are important lubricant rheological properties that affect the life span of machine elements. Similarly, temperature properties are useful in choosing lubricant for a predetermined purpose. Oils at elevated temperatures break down but in cold environment may solidify or even freeze. Therefore, the range of temperature for which the lubricant is effective is of utmost importance.

The rheological and temperature properties (viscosity at 40^0C, viscosity at 100^0C, viscosity index, pour-point temperature, cloud-point temperature and flash-point temperature) of the jatropha and castor oils were analysed in the Agricultural and Bioresources Engineering Laboratory and Central Teaching laboratories, FUT Minna. Details of the procedures are reported in the following subsections.

i. Measurement of viscosity

The dynamic viscosity was measured using the ASTM D2983 specification, using NDJ-5S (2013 model) digital viscometer that measures in the range 10 to $2X10^6$ mPas having an accuracy of $\pm$ 2 %. The rotor was cleaned using methanol and dried, then it was attached to the digital viscosity instrument and the oil to be measured was poured into a 30 ml sample bottle and the temperature was measured using a digital thermometer. The oil was mounted on the viscometer stand and the power button was turned on as shown in plate IV. After ten seconds the viscosity of the oil was displayed by the machine which was noted and recorded. The oil was then heated in a water bath to 40^0C and100^0C and the viscosities at both temperatures were measured

Plate IV: Measurement of dynamic viscosity

ii. Determination of viscosity index

The viscosity index is an indirect quantification of reduction in oil rheology as heat increases. elevated values shows that the oil experienced little viscosity decline relative to temperature. The viscosity index was determined by calculation using the following procedure and formula.

Initialy, oil kinematic rheology property is quantified at 40°C (which is called 'U') and at 100°C respectively. Then ASTM D2270 (ASTM, 1998a) was checked for the property at 100°C of the oil of interest, and the equivalent numbers for the reference oils, 'L' and 'H'. The obtained 'U', 'L' and 'H' were substituted in (3.5) to yield the viscosity index (V.I) as shown in equation (3.5).

$$\text{V.I} = \frac{(L-U)X100}{(L-H)} \tag{3.5}$$

iii. Measurement of cloud point and pour point

The pour point of oil is a useful property in the lubrication of any system in cold climate. If oil stops to flow it shows that enough wax crystallization has taken place or that highly viscous state the oil has been attained. At this stage waxes or high chemical mass paraffins precipitate out of the oil forming entangled solid particles preventing the remaining oil from flowing. This is a serious point since the successful operation of a machine relies on the continuous supply of oil to the parts in motion.

The cloud-point temperature was quantified according to the ASTM D2500 specification with the help of a refrigerator and digital infra-red thermometer with measuring range from -50^0C to 380^0C shown in plate V. The oil was first heated to 50^0C to ensure oil constituent is dissolved and elimination of any influence of past heat experienced. Heat was then extracted from it at a specific rate in the refrigerator and observed for the onset of wax precipitation in the form of a distinctive cloud or haze. The temperature at the onset of the cloudiness was

measured using the digital infra-red thermometer and written down as the cloud-point temperature.

The pour point was determined according to the ASTM D97 standard. The same procedure and equipment as that of the cloud point was employed. The decrease in the temperature of the oil at 3^0C was monitored while the test container was positioned to check for movement. The temperature 3^0C just before the point at which the oil stopped flowing was noted as the pour point.

Plate V: Digital infra-red thermometer

iv. Measurement of flash-point temperature

The flash-point temperature of the lubricating oil is the point at which its vapour will catch fire. Considering fire safety, ignition and fire points are very important, since they constitute the only factors which define the fire hazard of a lubricant. In general, the flash-point temperature of oils increases with increase in molecular weight.

The flash-point temperature was measured according to the ASTM D92 standard (ASTM, 2002c). 30 mls of the oil was poured into an open cup apparatus and heated at atmospheric pressure while the temperature was being monitored with the digital infra-red thermometer. The set up was carried out in a fume chamber where air was being supplied to the heated oil until it ignited. The temperature at which it ignited was noted and recorded as the flash point of the oil.

3.2.1.3 Determination of thermo-oxidative stability

Stability in the presence of oxygen (ASTM D943) measures resistance to chemical constituent scattering or rearrangement in air of a lubricant during temperature elevation (ASTM, 2004). Lubricants can undergo oxidation when left in air, especially at high temperatures. The service life of a lubricant is strongly controlled by this property. Thermal stability is lubricants resistance to chemical particles scattering or molecular rearrangement at increased heating level without oxygen interference.

The peroxide value is basically used as the basis for quantifying the stability of vegetable oils (Demian, 1990). The stabilities of the oils in the presence of heat and oxygen were determined in the WAFT laboratory by measuring peroxide values, the details of the measurements are reported as follows.

i. Determination of thermal stability

The resistance to thermal breakdown of the oils was determined from the peroxide value measurements carried out according the method adopted by Onuoha (2015). A control sample of the oil was left air tight in an inert environment while another sample was heated over an electric hot plate to smoke point (over 250^0C), then the heated sample was left to degenerate in an oxidizing environment for 14 days. After 14 days the peroxide value of the control

sample and the degenerated sample was measured and the difference in their peroxide values deducted from 100% was calculated and recorded as the thermal stability.

ii. Determination of oxidative stability (peroxide value)

The peroxide value is mostly a property of the classical oils and is a quantification of its oxygen; it is usually less than 10 mEQ O_2/kg (Codex, 1993). A taste of spoilage begins to show up between 20mEQO_2/kg and40 mEQ O_2/kg (Onuoha, 2015).

The peroxide value was measured based on AOCS C/8 53 specification where 0.001 kg oil was transferred to a clean drying and boiling tube, 0.001 kg of powdered iodide of potassium and 0.020 litres of solvent mixture (2 volume of glacial acetic acid + 1volume of chloroform) was added, the tube was boiled over water-steam fluid such that the liquid boils within 30seconds and was also left to boil with random intensity for just 0.5 minutes. The content was quickly transferred to a glass containing 20 ml of potassium iodide solution; and the tube cleaned out with 25ml of pure water, it was then made to react with 0.02 M sodium thiosulphate chemical in the presence of starch-indicator. The reaction of a blank was also done concurrently. The peroxide value (PV) was determined from equation (3.6)

$$\text{PV} = \frac{(A-B)XNX1000}{Woil} \qquad (3.6)$$

Where B= volume of sodium thiosulphate used in blank titration. A= volume of sodium thiosulpate used in titration with oil. N= normality of sodium thiosulphate (which is 0.02). Woil= weight of oil used.

3.2.1.4 Corrosion level test

Corrosion is a chemical reaction resulting in the conversion of the useful materials such as metals and their alloys into worthless oxides. The corrosion level of the oils was measured

using the procedure of Alves and Oliveira (2008), in Water Aquaculture and Fisheries (WAFT) laboratory, FUT Minna. This experiment was conducted to examine the number of corrosion spots that will form on a filter paper resulting from the corrosive action(s) of the plant oils and their derived lubricants. The experiment was done by measuring cast iron chips unto a filter paper inside a Petri dish. Then the oil of interest was collected with a pipette was used to wet the iron chips on the filter paper in the Petri dish and covered for 2 hours. After which the iron chips were discarded and the filter paper carefully rinsed out with tap water. The paper was treated with acetone and allowed to dry at room temperature, with the corrosion level assessed by sight.

3.2.1.5 Measurement of biodegradability

Biodegradability is the measurement of the level of biodegradation. Biological break down is the situation by which organic materials are disintegrated aerobically or anaerobically by the biological catalyst produced by micro-organisms. The term relates oftenly to environmental studies and contamination or environmental bioremediation (Aluyor *et al.,* 2009). The biodegradability test of the oils and lubricants as compared to that of the SAE20/W50 was carried out according to Ijah and Antai (2003). The test was carried out in the Microbiology Laboratory of FUT Minna. Sterile sampling bottles were used to collect water sample from Federal University of Technology Minna, Bosso campus drainage channel and soil sample was collected from petroleum oil contaminated soil in a motor cycle workshop in Dutse Kura Gwari area of Minna Niger State.

Microbes (bacteria and fungi) were isolated from the water and soil samples and identified by characterisation based on gram staining and biochemical tests. The oil samples were inoculated with the *Bacillus sp.* CDB-08 bacterial isolated from petroleum contaminated soil and the oils were recovered using diethyl-ether every seven days for 28 days. A control test was also carried out and the extent of the degradation was determined by gravimetric analysis

method. The method involved the extraction of the residual oils with 50 ml petroleum ether and noting its absorbance reading at 520 nm wavelength. The percentage biodegradability of the oils was also determined by weighing the amount of recovered oil at interval of 7 days over the period using equation (3.7).

$$\%\ biodegradation = \frac{(Wao - Wro)X100}{Wao} \quad (3.7)$$

Where W_{ao} = weight of the added oil; and W_{ro} = weight of the residual oil after days of bacterial inoculation.

3.2.1.6 Chemical transformation of vegetable oils

Some of the fastly biodegradable lubricating oils are from virgin unmodified oils. The triacylglycerol structure of vegetable oil makes it an excellent candidate for potential use as a base stock for lubricants and functional fluids. But, their thermo-oxidative and hydrolytic stabilities are unacceptable, their useless in cold weather behaviour that happens during serious temperature-pressure conditions and shear force and other tribochemical degrading processes also hinder their application as lubricant basestocks in their natural state (Srivastava & Sahai, 2013).

Therefore, virgin plant and animal oils are exclusively used in places having less heat stress. An attractive way of removing these problems is Chemical transformation of plant-seed oils (Asadauskas & Erhan, 1999). Chemical transformation is necessary to improve these engineering limitations so as to eliminate the bis allelic hydrogen activity in methylene disrupted the many-unsaturation, and to optimise the extend of chemical distortion for better cold weather application (Erhan & Asadauskas, 2000; Kalhapure *et al.,* 2015). To avoid the problem of soap forming, a two-stage procedure was employed to chemicaly transform Jatropha curcas oil and Ricinus communis oil into esterified oils. The following subsection

reports the details of chemical modification that was carried out on the jatropha and castor oils.

The oils were modified by acid catalysed esterification treatment which is looked at as a treatment before the real reaction of the crude oils done for bringing down their water content. soap formation and subsequently, reduction in free fatty acid (FFA) content is predominantly caused by water content (Ho *et al*., 2020). The acid catalysed esterification was done according

the method used by Abdulkareem *et al*., (2012) and Salimon *et al.,* (2014) but with slight changes. The oil was fired up in the glass flask to 60^{o}C and a liquid sulphuric acid (1 out of 100 parts based on the oil volume) in methanol (3 out of 10 parts by volume) fired up to 45^{o}C and introduced into the reaction flask. The resultant fluid was placed in a water bath heated to 60^{o}C and stirred with a mechanical stirrer for 1.5 hours as shown in plate VI. The content was then poured into a separating funnel and allowed to settle for 24 hours. The methanol-water fraction at the top layer was removed and the oil was decanted and washed with distil water until a pH of 7.0 was achieved as shown in plate VI.

(a) (b) (c)

Plate VI: Esterification of jatropha and castor oil (a) oil in a reactor inside water bath (b) preheating of the water bath (c) castor and jatropha oil esterification products in separation funnels

3.2.2 Development of jatropha and castor oil based biolubricants

The modified jatropha and castor oils were used to develop biolubricants using a two stage transesterification reaction. Details of the development are reported in sections 3.2.2.1 and 3.2.2.2.

3.2.2.1 Synthesis of jatropha and castor oils methyl esters

The methyl esters were synthesised from the esterified oil which were having a percentage free fatty acid less than 1%. The dried methyl esters are the beginning materials for the synthesis of vegetable oil based biolubricant. The synthesis of the methyl esters was carried out according to Talib *et al*., (2017) and Kadry (2015).

The esterified oil was heated at 130^0C for 30 minutes to boil out. The dried esterified oil was charged into a 500 ml conical flask reactor and heated to 60^0C in a water bath. 1 % w/w Potassium hydroxide (KOH) was weighed and dissolved in methanol. The alkanol to oil molar ratio of 10 is to1 was used and the entire mixture added into the already hot oil in the reactor inside the water bath maintained at 60^0C as shown in plate VII. The reactants were stirred with a Grant mechanical stirrer at 600 rpm speed for 90 minutes, after which the product of the reaction was allowed to cool in a separating funnel for 24 hours. The lower layer which consists of glycerol excess methanol catalyst and other impurities was separated out of the funnel the upper layer made up of methyl ester, methanol, residual catalyst and impurities were purified by washing with distilled water at 70^0C until the washing water had a pH of 7.0 as shown in plate VII. The remaining water in methyl ester was removed by heating in open air at 80^0C.

3.2.2.2 Development of jatropha and castor oils biolubricants from methyl esters

The castor oil and jatropha oil biolubricants were produced using their synthesised dry methyl esters. The production of the biolubricants was done in accordance to Musa *et al*., (2015) and Musa *et al*., (2016) but with slight changes. The catalyst which is Potassium methoxide (30% w/w KOH in methanol) was prepared by weighing out the KOH and dissolving it in methanol at 60^0C. Then 368 grams (400 mls) of the methyl ester was measured into a three-neck flask equipped with a digital thermometer, magnetic stirrer and heater positioned in a fume chamber for extraction of methanol as by product as shown in plate VIII.

(a) (b) (c)

(d) (e)

Plate VII: Transesterification to produce methyl ester (a) separation of methyl ester from glycerol (b) water bath heater and stirrer (c) washing of methyl easter (d) drying of methyl ester (e) methyl ester final product

The methyl ester was fired up to 60^0C after which trimethylolpropane (TMP) was measured (mole ratio of methyl ester to TMP is 4.0: 1) and added into the reactor. 0.8% w/w of the prepared KOH catalyst was then added to the reactor and the temperature was raised to 120^0C and stirred at 600 rpm for three hours. The biolubricants were produced in batches of 400 mls and the fume chamber was switched on throughout the production to extract the produced methanol and reduce the excessive foam formation.

(a) (b) (c)

Plate VIII: Development of jatropha and castor oils biolubricants (a) Experimental setup in fume chamber (b) Powered magnetic heater and stirrer (c) Digital thermometer displaying reaction temperature

i. Washing and drying of castor and jatropha oils based biolubricants

At the end of three hours when the reaction for the production of biolubricant was completed, the reaction products were transferred into a funnel-like flask to cool and settle under for 24 hours. Then the produced biolubricant was washed with clean water having 10% v/v of O-phosphoric acid (35%) in a separating funnel to remove catalyst. This was followed with continuous washing with pure (distilled) water at 70^0C until 7.0 pH solution was gotten, then it was dried over a hot plate as shown in plate IX.

(a) (b) (c)

Plate IX: Separation and washing of Jatropha and castor oil biolubricants (a) Separation of biolubricant from by-products (b) Washing of biolubricant (c) Cleanly separated biolubricant and water

ii. Jatropha and castor oils biolubricants confirmation tests

The castor oil and jatropha oil biolubricants produced were analysed using Fourier Transform Infra-red (FTIR) spectroscopy to confirm the formation of castor oil ester TMP and jatropha oil ester TMP. Gas chromatography (GC-MS) analysis was also carried out on the biolubricants to deteremine the percentage composition of the TMP esters. The physicochemical, rheological, temperature and thermal properties of the biolubricants were also determined.

The FTIR spectroscopy is a technique used to identify organic and inorganic compounds in a sample. The system was powered and after warming up for 10 minutes, about 15 mg of the sample was placed on the equipment and properly aligned. Then the wavelength as well as

the transmittance and absorbance of the sample was read from the connected computer system and recorded.

The GC-MS confirmation tests informs us about the product chemical components of the biolubricant (fatty acid (FA), monoester (ME), diester (DE) and trimester (TE). The GC- MS analysis was carried out at the Multiuser Laboratory ABU Zaria using Mass Hunter GCMS machine. The following machine conditions specified by the manufacturer for detection of organic esters were employed: Column flow was 1.80mL/min Column over temperature of 70.0° C; pressure of 116.9 kpa and flow of 40.8mL/min at linear velocity of 49.2cm/sec.

3.2.3 Tribological evaluation of oils and developed lubricants

The tribological evaluation of the oils and developed lubricants was achieved in the Metallurgical and Materials Engineering laboratory of Ahmadu Bello University Zaria. The evaluation was done using the Anton Paar standard tribometer TRN version 6.1.19.

Circular 10 mm diameter aluminium (SAE 332) disc was prepared. The disc has tensile strength of 248 MPa, yield strength of 193 MPa and 1% elongation at 24^0C. The composition of the circular disc is shown in Table 3.4. The disc was mounted on the rotating plate holder as shown in Figure 3.1. Then 2 mls of the lubricant was added on to the disc and ball contacting surface and the system was powered and the tribo-test parameters shown in Table 3.5 were entered into the system. The tribometer was turned on for data acquisition, the acquired data was saved for further processing at each experimental interval.

Table 3.4: Chemical Composition of the SAE 332 Aluminium circular disc

Element	Si	Cu	Mg	Al
% composition	9.5	3.0	1.0	86.5

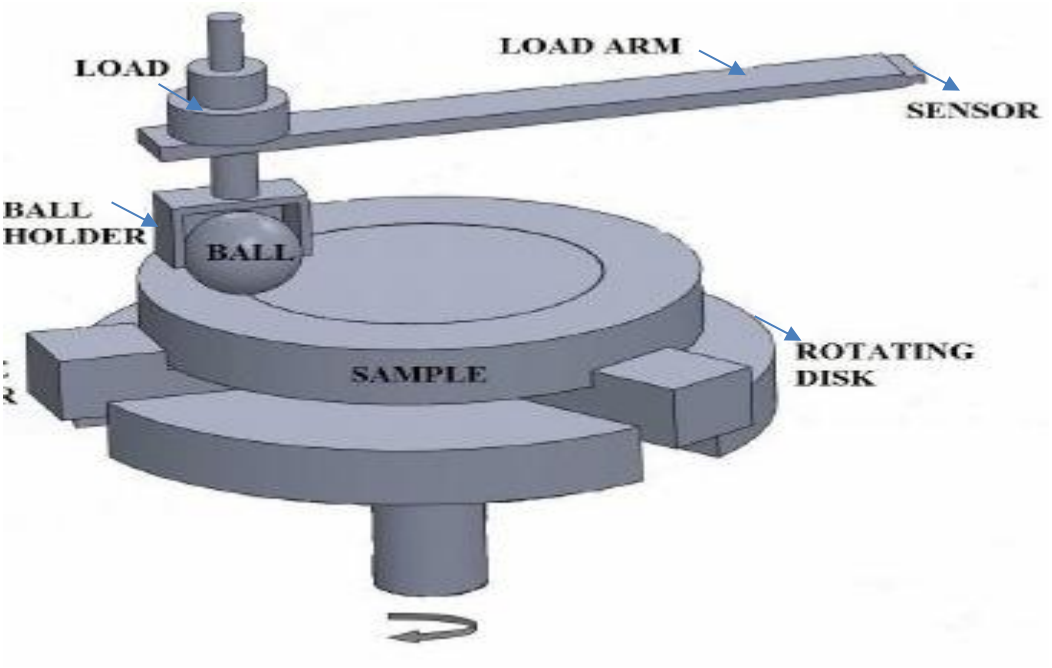


Figure 3.1: Ball on disc tribometer (modified from Alemdag *et al.,* 2015)

Table 3.5: Tribotester parameter for tribological evaluation

S/N	Parameter	Unit	Value
1	Dimension of ball	mm	6.0
2	Diameter of disc	mm	30.0
3	Track radius	mm	5.0
4	Linear speed	cm/s	10.0
5	Normal load	N	8.0
6	Run time	s	1500.0
7	Linear distance	m	150
8	Acquisition rate	Hz	10
9	Lubricant volume	ml	2.0

3.2.4 Determination of selected additives with dominant effect on tribological performance of developed lubricants

To achieve the fourth objective a statistical design of experiment was used followed by the formulation, blending and testing of the vegetable oil based lubricants and additive blends. Details of the design and formulation are outlined in sections 3.2.4.1 and 3.2.4.2.

3.2.4.1 Design of experiment to determine the most significant additive

Design of experiment procedures are key applications in engineering design activities, whenever new products are developed and existing ones are made better. Statistical design of experiment refers to the procedure of planning the experiment so that needed data is collected and analysed using statistical methods. Statistical design of experiments enables the researcher to make valid and objective conclusions.

This study optimised various concentrations of additives which included: concentration of anti-wear additive, concentration of viscosity modifier, concentration of anticorrosion additive and extreme pressure additive in lubricants. The Taguchi method was used to determine influence of the control parameters on the coefficient of friction, viscosity index and wear rate. Minitab 17 computer software was used for choosing the orthogonal array. The experimental factors and coded levels used in the design of the experiment are shown in Table 3.6 while the actual selected values of the levels are shown in Table 3.7.

The Taguchi method of design of experiment was used, three level orthogonal $L_9(3)^4$ array was used for the design. The experimental runs are displayed in Table 3.8. The total of 36 lubricant samples were blended, 9 each from unmodified jatropha oil, unmodified castor oil, jatropha biolubricant and castor biolubricant.

Table 3.6: Factors and coded levels taken into account while planning experiments

Factors	Level I	Level II	Level III
Antiwear additive (% w/w)	-1	0	1
Viscosity modifier (% w/w)	-1	0	1
Extreme pressure additive (%w/w)	-1	0	1
Anticorrosion additive (% w/w)	-1	0	1

Table 3.7: Factors and levels taken into account while planning experiments

Factors	Level I	Level II	Level III
Antiwear additive “A” (% w/w)	1	2	3
Viscosity additive “B” (% w/w)	1	2	3
Extreme pressure additive “C” (%w/w)	0.3	0.6	0.9
Anticorrosion additive “D” (% w/w)	2	2.5	3

Table 3.8: Designed experimental runs with coded values

Trial	Antiwear additive	Viscosity additive	Extreme pressure additive	Anticorrosion additive
1	-1	-1	-1	-1
2	-1	0	0	0
3	-1	1	1	1
4	0	-1	0	1
5	0	0	1	-1
6	0	1	-1	0
7	1	-1	1	0
8	1	0	-1	1
9	1	1	0	-1

3.2.4.2 Formulation, blending and testing of vegetable oil-based lubricants

To formulate the lubricants, the coded values of the experimental runs were replaced with % weight composition of the additives as shown in Table 3.9. Total weight of 50 g of lubricant was formulated for each run.

Table 3.9: Designed experimental runs with % w/w composition of additives

Run	A (% w/w)	B (% w/w)	C (% w/w)	D (% w/w)
1	1	1	0.3	2
2	1	2	0.6	2.5
3	1	3	0.9	3
4	2	1	0.6	3
5	2	2	0.9	2
6	2	3	0.3	2.5
7	3	1	0.9	2.5
8	3	2	0.3	3
9	3	3	0.6	2

Oil and additives were weighed and mixed in a 100 ml conical flask. Thus 50 g of lubricant was prepared for run 1 as follows:

1) 1% of anti-wear additive (A) in 50 g lubricant = 0.5 g
2) 1% of viscosity additive (B) in 50 g lubricant = 0.5 g
3) 0.3% of extreme pressure additive (C) in 50 g lubricant = 0.15 g
4) 2% of anticorrosion additive (D) in 50 g lubricant = 1 g
5) Total weight of additives (0.5+0.5+0.15+1) = 2.15 g of additives
6) Balance of oil in lubricant (50-2.15) = 47.85 g of oil

The weights for each run were determined and tabulate as shown in Table 3.10. For each run the calculated oil and additives were weighed and mixed in the 100 mls capacity flask. Then the mixture was blended over a magnetic stirrer and heater at 70^0 C and 600 rpm for 10 minutes. The developed lubricants at the end of blending were allowed to cool to room temperature and taken to the laboratory for tribological evaluation.

Table 3.10: Designed experimental runs with mass (g) composition of additives and oil

Run	A (g)	B (g)	C (g)	D (g)	Total additive (g)	Balance oil (g)
1	0.5	0.5	0.15	1.00	2.15	47.85
2	0.5	1.0	0.30	1.25	3.05	46.95
3	0.5	1.5	0.45	1.50	3.95	46.05
4	1.0	0.5	0.30	1.50	3.30	46.70
5	1.0	1.0	0.45	1.00	3.45	46.55
6	1.0	1.5	0.15	1.25	3.90	46.10
7	1.5	0.5	0.45	1.25	3.70	46.30
8	1.5	1.0	0.15	1.50	4.15	45.85
9	1.5	1.5	0.30	1.00	4.30	45.70

3.2.4.3 Determination of the contributions of additives

The empirical data collected were analysed using analysis of variance (ANOVA) to determine the percentage contributions of each of the additives on the tribo-performance of the developed biolubricants. Moreover, the results were presented using normal probability plots. The ANOVA table was calculated from equations 3.8 to 3.10. The percentage contribution (P) of each of the input factors is calculated using:

$$P = SS_{individual}/SS_{Total} \quad (3.8)$$

Where, SS is the sum of squares

$$SS = \sum_{i=1}^{n} {y_1}^2 - \frac{1}{n}\sum {y_1}^2 \quad (3.9)$$

Such that n is the conducted experiments total, and y_1 is the response

$$SSerror = SS_{Total} - (\text{sum of individual values of SS}) \quad (3.10)$$

The effects of the interactions of the additives upon the tribo-performance of the biolubricants was also researched using interaction plots. The amount of additive required to

impact desirable tribological performance on the developed biolubricants was determined using contour plots and 3-D surface plots.

CHAPTER FOUR

4.0. RESULTS AND DISCUSSION

4.1 Characterisation of Jatropha and Castor Oils

The results and discussion of the result of the characterisation of the jatropha and castor oil are presented in the following subsections.

4.1.1 Physicochemical characterisation of jatropha and castor oils

The physical and chemical behaviour of castor and jatropha oils is shown in Table 4.1. From the result jatropha oil has a specific gravity of 0.913 and density of 913 kg/m^3, which is slightly higher than the values (0.903 and 903 kg/m^3) reported by Akbar *et al.,* (2009). Similarly, the specific gravity and density of castor oil is 0.955 and 955 kg/m^{3}respectively. These values for castor oil are slightly lower than the ASTM standard range (0.957 -0.961) The slight differences are as a result of differences in climate and soil conditions where the plants were grown and the condition of test. It also can be seen that the castor oil is denser than the jatropha oil while both oils are less dense compared to water and would therefore float in water.

The acid number of the jatropha oil is 31.15 mg KOH/g and its percentage free fatty acid is 15.6 and that of the castor oil is 39.48 mg KOH/g and 19.74 respectively. These values are too high and indicate that the two oils are non edible as the triglycerides in the oils have been decomposed. The lower the acid number the better the oil as a lubricant as the high acid number oils are likely to corrode and wear machine parts that are lubricated. The results show that the castor oil is a worse lubricant compared to the jatropha oil with regards to the acid value. Both oils will need modification to bring down their acid value to be better industrial lubricants.

The soap forming tendency number of an oil measures tendency of the oil to turn into soap during the organic neutralisation. The Jatropha oil and castor oil saponification value gotten

were seen to be 220.46 mgKOH/g and 185.41mgKOH/g respectively. These are slightly outside the AOCS specifications for the oils. These high saponification values shows that the oils at their natural state will be more suited for soap and cream making than for use as a lubricant, thus it may be necessary to modify the oils before use as lubricants. The soap forming tendency number of Jatropha oil (220.46 mgKOH/g) exceeded that of castor oil (185.41mgKOH/g) and thus signifies that jatropha oil has higher tendency to form soap during organic neutralisation than the castor oil.

Table 4.1: Physicochemical Properties of Jatropha and castor Oils

S/No.	Parameter	Jatropha oil	Castor oil	SAE 20/W50
1	Specific Gravity	0.913	0.955	0.878
2	Free Fatty Acid (mg KOH/g)	15.6	19.74	-
3	Saponification Value (mg KOH/g)	220.46	185.41	-
4	Acid value (mg KOH/g)	31.15	39.48	-
5	Iodine Value (gI_2/100g oil)	88.9	92.1	80
6	pH	5.82	5.76	7.12

The measure of saturation level using iodine depicts unsaturation extend of the oil and also influences the rate and quantity of oxidation and deposition formed in internal combustion engines. The drying property of the oil is also determined using iodine value. The measure of saturation level using iodine for both Jatropha and castor oils gotten were observed to be 88.9 gI_2/100 g and 92.1 gI_2/100 g respectively. The Jatropha iodine value of (88.9 gI_2/100 g) was noted to be lower than castor oil iodine value (92.1 gI_2/100 g) and this signifies that there is a higher degree of unstauration in the castor oil than in the jatropha oil, Thus, both oils are classified as non-drying oils since their iodine value is below 115 gI_2/100 g.

The pH value is a quantification of acidity-level or alkalinity-level of a fluid. The pH of the jatropha oil was 5.82 while that of the castor oil was 5.76, thus both oils are acidic but the castor oil is more acidic. It is more desirable for a lubricant to have a pH between 8.0 and 10.0; lubricant with very low or too high pH can be damaging to the skin of the end users. Besides microbial deterioration of biolubricants takes place in acidic medium rather than alkaline medium. The acidic pH of the jatropha and castor oils will reduce the corrosion protection of the machine components being lubricated thereby reducing their life span thus it is necessary to modify these oils to be suitable for use as lubricants.

4.1.2 Rheological and temperature properties of jatropha and castor oils

The rheological properties (viscosity and viscosity index) of the vegetable oils are shown in Table 4.2; while the temperature properties (cloud point, pour point and flash point) are shown in Table 4.3. Kinematic viscosity is one of the deciding parameters to evaluate the effectiveness of a lubricant. An increase in kinematic viscosity means increase in the lubricating property of the fluid (Rao *et al.,* 2007).

From the results jatropha oil has a kinematic viscosity of 83.2cSt at 40^0C while oil from castor bean has kinematic viscosity at 40^0C of 280.6 cSt. The kinematic rheology of the castor oil is over three times that of the jatropha oil and it implies that jatropha oil has advanced flow capability compared to castor oil, thus, it will easily undergo organic neutralisation. However, the castor oil viscosity at 100^0C (77.5cSt) was only slightly higher than that of jatropha oil at 100^0C (63.5cSt). This means that the jatropha oil has a higher viscosity index than the castor oil, therefore the jatropha oil will show less variation in viscosity at high temperatures compared to the castor oil. The jatropha oil confirms to ISO VG 68 grade of industrial oil while the castor oil confirms to the ISO VG 220 grade of industrial oil.

Table 4.2: Rheological Properties of Jatropha, castor and Mineral Oils

S/No.	Parameter	Jatropha oil	Castor oil	SAE 20/W50
1	Viscosity at 40^0C (cSt)	83.2	280.6	236.9
2	Viscosity at 100^0C (cSt)	63.5	77.5	99.1
3	Viscosity Index	145.5	33.4	65
4	Dynamic Viscosity at 40^0C (mPas)	76	268	208
5	Dynamic Viscosity at 100^0C (mPas)	58	74	87

The cold flow (pour point) of the oil from castor bean (-23.2^{0}C) is fantasticaly low which is a desirable property, the castor oil has a lower pour point than that of the jatropha oil (-11.2^{0}C). Both the jatropha and castor oils have good cold flow properties (cloud point and pour point) which have been what is lacking in most vegetable oils that hinder their applications in systems exposed to low temperatures. Thus, jatropha and castor oils can be used for lubrication of machines operating in cold climate such as automotive engines, construction machines, military equipments and space applications.

The flash point of the castor oil (282^{0}C) is higher than that of the jatropha oil (264^{0}C). Both oils have very high flash points; a lubricant desirable characteristic from the safety point of

view. Castor and jatropha oils are safer to be deployed at high temperatures as lubricants than most lubricating oils with flash point of 210^0C and fire point about 230^0C (Stachowiak, & Batchelor, 2000). Thus, it is safer to use jatropha or castor oil as lubricants in applications where there is tendency for accidents.

Table 4.3: Temperature Properties of Jatropha and castor Oils

S/No.	Parameter	Jatropha oil	Castor oil	SAE 20/W50
1	Pour point (^{0}C)	-11.2	-23.2	-24.1
2	Cloud point (^{0}C)	-8.3	-12.4	-18.9
3	Flash point (^{0}C)	264.0	282.0	255.0

4.1.3 Thermo-oxidative stability characterisation of jatropha and castor oils

The peroxide value is the usual method of assessment of primary oxidation products. Peroxide value of any oil gives an indication of its oxidative and thermal stability. The peroxide value of the jatropha is 5.98 meq/kg and peroxide value of castor oil is 8.92 meq/kg while the peroxide value of SAE 20/50 is 0.99 meq/kg.

The peroxide value of jatopha oil (5.98 meq/kg) is lower than the peroxide value of castor oil (8.92 meq/kg), thus jatropha oil has a better oxidative and thermal stability compared to castor oil. The castor oil has lower oxidative stability than the jatropha oil because it is more unsaturated than the jatropha oil. This concurs with the findings of other scholars like Aravind *et al.,* (2018). However, these peroxide values of the jatropha and castor oils are too high showing that the oils have poor thermo-oxidative stabilities.

The outcome agrees with the findings of all other researchers that had studied the thermo-oxidative stabilities of animal and plant oils (Gunstone, 2004). The poor stability in the presence of heat and oxygen of the jatropha and castor oils implies that lubricants formulated

from these two oils will have a low shelf life as degradation of the oils will take place very fast. The poor thermo-oxidative stability of castor and jatropha oils are a major hindrance for their application as industrial lubricants, thus the oils must be modified for them to be useful in the production of industrial lubricants.

4.1.4 Corrosion inhibition properties of jatropha and castor oils

Plate X (a) shows the humidified cast iron chippings on filter paper covered in a petri dish while Plate X (b) is the jatropha oil (JO) filter paper after 2 Hrs and Plate X (c) shows the castor oil (CO) filter paper after 2 Hrs. No rust spot was found on both filter papers containing cast iron soaked in jatropha oil (JO) and castor oil (CO). Thus, both JO and CO exhibit excellent corrosion inhibition characteristics and based on Alves and Oliveira (2008), both jatropha oil and castor oil are of corrosion grade 0.

(a) (b) (c)

Plate X: Corrosion inhibition tests of jatropha and castor oils (a) Test with cast iron particles, (b) after 2Hrs, JO, (c) after 2Hrs, CO

4.2 Modification of Jatropha and Castor Oils

The modification of the jatropha oil (JO) produced 95% yield of esterified jatropha oil (EJO), while the esterification of the castor oil (CO) produced 90% yield of esterified castor oil (ECO). The castor oil had a lower yield compared to the jatropha oil because it contained more impurities that are gum like which remained on the body of the glass reactor as shown in plate XI. The modified oils were also characterised to see the influence of the modification on the physicochemical, rheological, temperature and corrosion inhibition properties of the oils. Details of the results are presented in the following subsections.

(a) Before (b) after

Plate XI: Reactor before (a) and after (b) esterification of castor oil

4.2.1 Fourier transform infrared (FTIR) imaging of modified jatropha and castor oils

The modification of the jatropha triglycerides to EJO and modification of castor triglycerides to ECO was confirmed using FTIR. The FTIR absorption image of the EJO is shown in Figure 4.1 while the result of the ECO is shown in Figure 4.2.

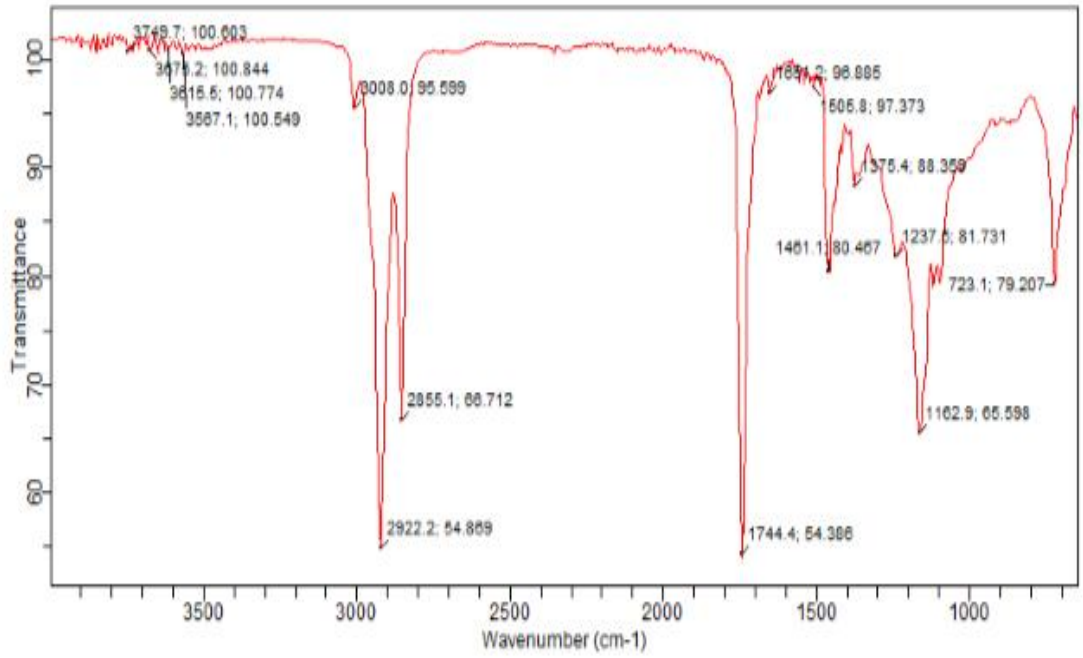


Figure 4.1: Esterified jatropha oil (EJO) FTIR spectra

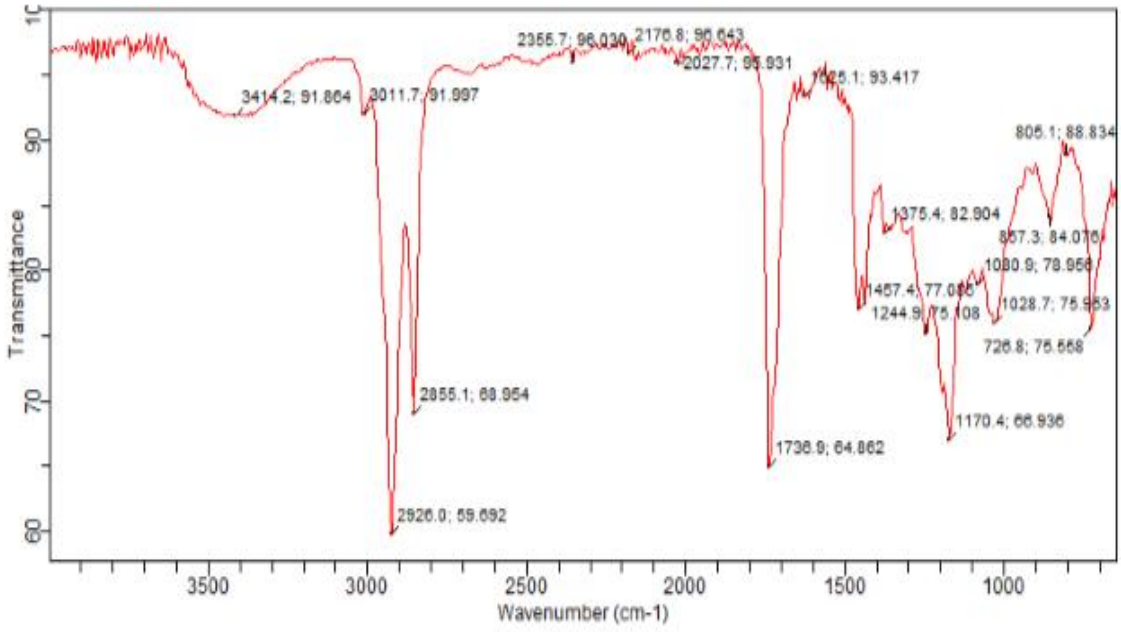


Figure 4.2: Esterified castor oil (ECO) FTIR spectra

From the spectra of the EJO shown in Figure 4.1, the peak of the ester can be seen at a wave length of 1744.4 cm^{-1}. Also, from the spectra of the ECO displayed in Figure 4.2 the presence

of the ester can be seen in the peak at the wave number 1736.9 cm^{-1}. The peaks in the two spectra confirm the modification of the castor and jatropha oils into esterified castor oil and esterified jatropha oil respectively.

4.2.2 Physicochemical properties of modified jatropha and castor oil

The chemical and physical characteristics of the esterified jatropha and castor oils are shown in Table 4.4. The specific gravity of the ECO is 0.931 and its density is 931 kg/m^3 while that of the modified jatropha oil is 0.908 and 908 kg/m^3 respectively. The modified castor oil is denser than the modified jatropha oil. The modified oils have lower density compared to the unmodified oils, therefore, esterification of jatropha and castor oils reduces their density.

Table 4.4: Physicochemical Properties of modified Jatropha and castor Oil

S/No.	Parameter	Esterified Jatropha oil (EJO)	Esterified Castor oil (ECO)	SAE 20/W50
1	Specific Gravity	0.908	0.931	0.878
2	Free Fatty Acid (mg KOH/g)	0.465	0.898	-
3	Saponification Value (mg KOH/g)	214.12	192.5	-
4	Acid value (mg KOH/g)	0.931	1.80	-
5	Iodine Value (gI_2/100g oil)	87.6	88.9	80
6	pH	7.12	4.3	7.12

The acid number of the ECO (1.80 mgKOH/g) is higher than the acid number of the EJO (0.931 mgKOH/g), similarly the free fatty acid content of the ECO is higher than that of the EJO. Generaly, the modification of the two oils improved the lubricant properties of the oils because the acid number and free fatty acid contend of the oils were reduced drastically by esterification. The reduction in acid number of the oils through esterification indicates a reduction in the tendency of the modified oils to corrode machine parts that are being lubricated.

The saponification value of the modified jatropha oil is 214.12mgKOH/g. This is greater than that of the modified castor oil (192.5 mgKOH/g). This indicates that the modified jatropha oil has a higher tendency to form soap more than the modified castor oil. The modification of the jatropha oil reduced the saponification value from 220.46 to 214.12 mgKOH/g. This reduction in saponification value improved its characteristics for use as feedstock for industrial lubricants. The modification of the castor oil on the other hand slightly increased its saponification value from 185.41 to 192.5 mgKOH/g, which is not a desirable change with regards to being a feedstock for industrial lubricants.

The iodine value of the modified jatropha oil (87.6 g I_2/100g) is slightly lower than that of the modified castor oil (88.9 g I_2/100g). Modification of the oils reduced their iodine value which shows that degree of the oils unsaturation were reduced by transesterification. Therefore, the stability in the presence of heat and oxygen of the oils is improved by the modification. Both EJO and ECO are non-drying oils as their iodine values are less than 115 I_2/100g.

From the result, the pH of the EJO is 7.12 which is alkaline, while that of the ECO is 4.3 which is acidic. While modification of jatropha oil increased its pH from 5.82 to 7.12 the medication of castor oil reduced its pH from 5.76 to 4.3. The EJO is a better lubricant

feedstock than the ECO because it is alkaline unlike the ECO that is acidic and will encourage corrosion in machine parts.

4.2.3 Rheological and thermal properties of modified jatropha and castor oil

The kinematic rheology (viscosity) of the modified jatropha oil when compared with that of jatropha oil and that of modified castor oil as compared to that of castor oil is shown in Figure 4.3. From the observed outcome it is seen that modification of jatropha oil improved its viscosity at 40^0C from 83.2cSt to 96.9 cSt. The viscosity of the jatropha oil at 100^0C was however reduced from 63.5cSt to 47.4 cSt by the modification. The viscosity index of the jatropha oil was improved from 145.5 to 149.3 by the modification.

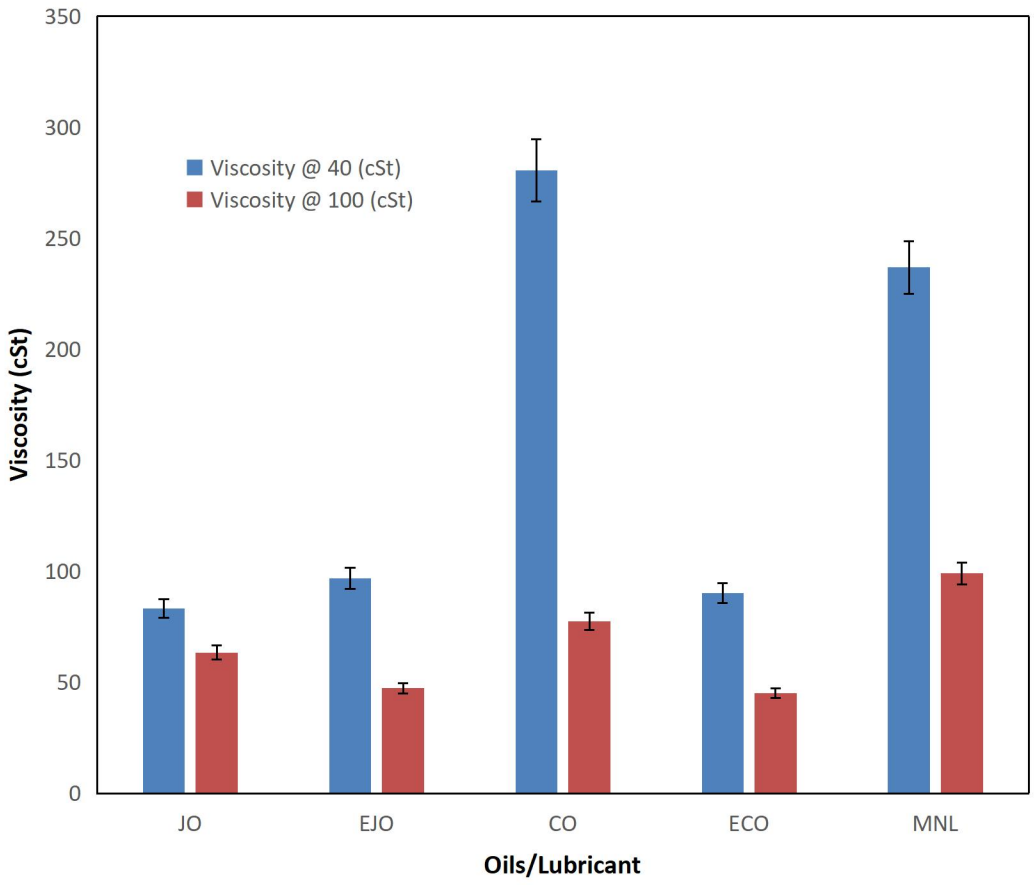


Figure 4.3: Kinematic viscosity of modified, unmodified vegetable oils and mineral oil lubricant

For the castor oil, modification drastically reduced the viscosity at 40^0C from 280.6 cSt to 90.2 cSt, and the viscosity at100^0C from 77.5 cSt to 45.1 cSt. However, the viscosity index was highly enhanced. The drastic reduction in viscosity at 40^0C for castor oil after modification is as a result of the removal of the gummy impurities in the oil by esterification. Comparing the oils with the commercially available mineral oil base lubricant SAE 20/W50 (MNL), it was found that the castor oil viscosity at 40^0C is more in number compared to the petroleum oil lubricant, contrary-wise JO, EJO and ECO has viscosity at 40^0C and viscosity at 100^0C that is lower than that of the mineral oil based lubricant.

The temperature behaviour (cloud-point temperature, pour-point temperature and flash-point temperature) of the modified oils are shown in Figure 4.4. Whereas both jatropha and castor oils have excellent cold flow characteristics (cloud and pour points), modification of the oils by esterification enhanced their cold flow behaviour. The cloud point of jatropha oil was improved from -8.3 to -8.7^0C and the pour point from -11.2 to -13.2^0C. Similarly, the cloud point of castor oil improved from -12.4^0C to -14.1^0C and its pour point from -23.2^0C to -27.5^0C.

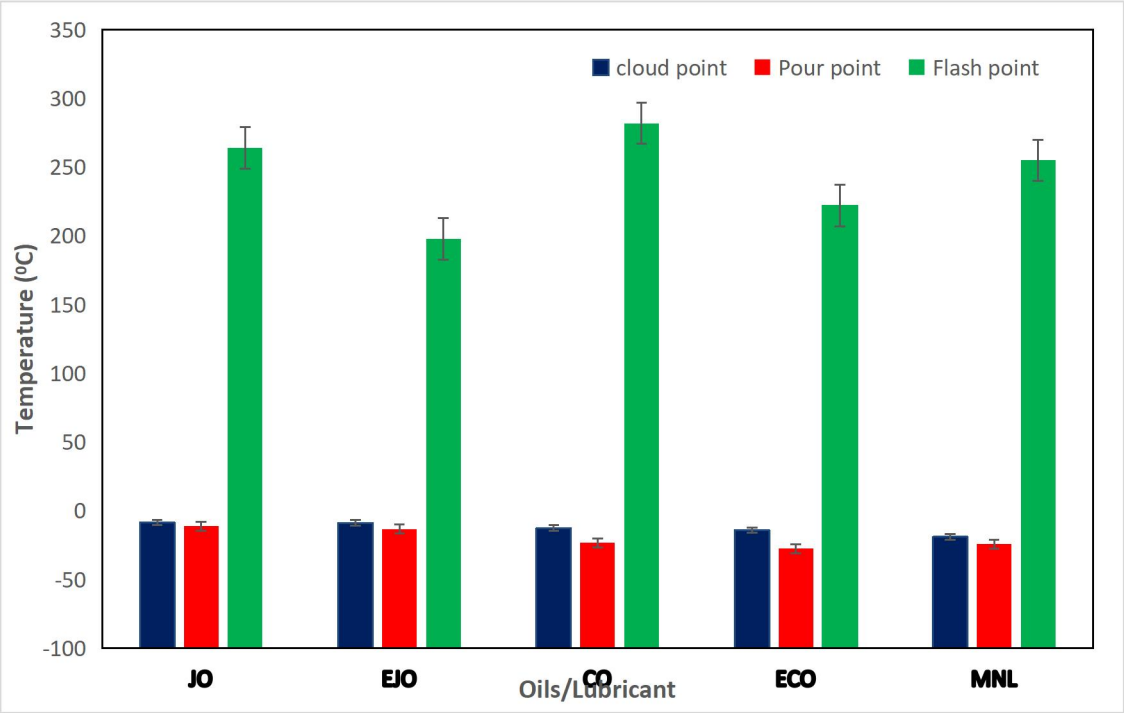


Figure 4.4: Temperature properties of vegetable based oils and mineral lubricant

The modification of the oils reduced their flash points; the flash point of the jatropha oil reduced from 264 to 198^{0}C and that of the castor oil from 282 to 222^{0}C. Both castor oil and jatropha oils have flash points greater than that of the mineral oil (MNL), however the flash point of MNL is higher than that of both modified jatropha and castor oils. The cloud point of the mineral oil is better than that of the modified and unmodified jatropha and castor oils. The pour point of the modified castor oil is superior to the pour point of the mineral oil.

4.2.4 Oxidation and thermal stability of modified vegetable oil

The peroxide value is the usual method of assessment of the primary oxidation products. Peroxide value of any oil gives an indication of its thermo-oxidative stability. The peroxide value of the modified jatropha oil was 3.99 meq/kg, for the modified castor oil was 1.99 meq/kg and that of commercial mineral oil lubricant SAE 20W50 was 0.99 meq/kg. From the results, the peroxide value of the jatropha oil after modification improved from 5.98 to 3.99 meq/kg while that of the castor oil greatly improved from 8.92 to 1.99 meq/kg. The modified castor oil has a lower peroxide value compared to the modified jatropha oil hence, the modified oil from castor bean had better thermo-oxidative stability than modified jatropha oil. All the vegetable oils both modified and unmodified had a peroxide value higher than that of the commercial petroleum fluid (MNL) SAE 20/W50. Modification of jatropha and castor oils improved their thermo-oxidative stability.

4.2.5 Corrosion inhibition of modified jatropha and castor oils

Plate XII (a) shows the humidified cast iron chippings on filter paper covered in a petri dish while Plata XI (b) is the modified jatropha oil (EJO) filter paper after 2Hrs and Plate XII (c) shows the modified castor oil (ECO) filter paper after 2Hrs. No rust spot was found on both filter papers containing cast iron soaked in modified jatropha oil (EJO) and modified castor oil (ECO). Thus, both EJO and ECO exhibits excellent corrosion inhibition characteristics

and based on Alves and Oliveira (2008), modified jatropha oil and modified castor oil are of corrosion grade 0.

(a) (b) (c)

Plate XII: Corrosion inhibition characteristics of modified jatropha and castor oils (a)Tests with cast iron particles, (b) after 2Hrs, EJO, (c) after 2Hrs, ECO

4.3 Development of Jatropha and Castor Oil Biolubricants

The jatropha oil biolubricant (JBL) produced had 96.95% yield of jatropha TMP esters (JBL), while the transesterification of the castor oil methyl ester (CME) produced 96.56% yield of castor oil TMP esters (CBL). The biolubricants (JBL and CBL) were produced based on optimum conditions documented in literature (Musa, *et al.,* 2015 and Musa, *et al.,* 2016). The biolubricants produced were analysed and details of the result are presented in the following subsections.

4.3.1 Fourier transform infrared (FTIR) imaging of jatropha and castor oil biolubricants

The presence of ester TMP in the produced biolubricant was confirmed using FTIR analysis. Figure 4.5 is the spectra of the jatropha oil biolubricant (JBL) while Figure 4.6 is the spectra of the castor oil biolubricant produced

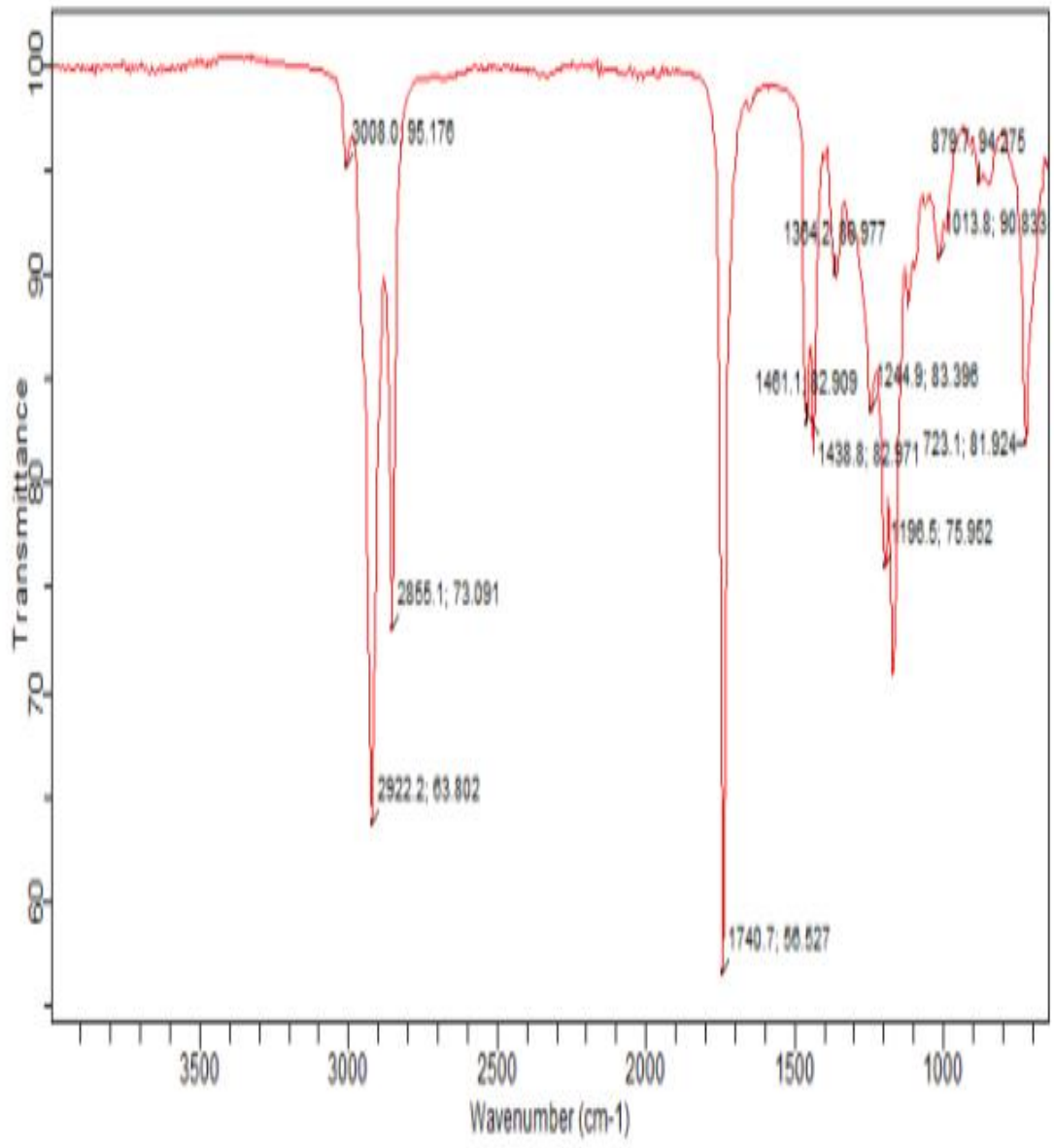


Figure 4.5: Jatropha oil biolubricant (JBL) FTIR spectra

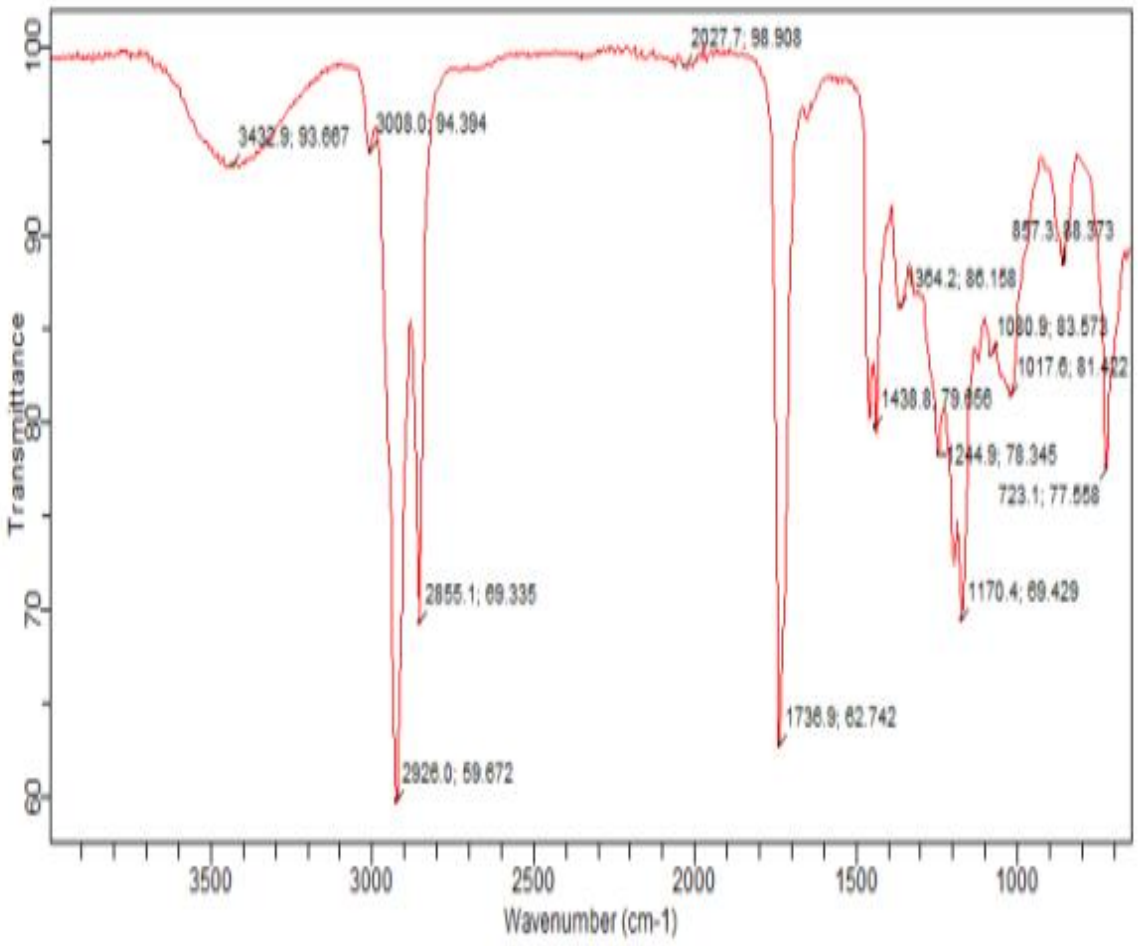


Figure 4.6: Castor oil biolubricant (CBL) FTIR spectra

. The presence of jatropha TMP ester is found at the peak with wave number 1740.7 cm^{-1} while the presence of castor oil ester TMP is confirmed at the peak of wave number 1736cm^{-1}. In Figure 4.6 the castor oil biolubricant also has a peak showing the retention of the hydroxyl group at 3432.9cm^{-1}wave number, however this hydroxyl group is absent from the jatropha oil biolubricant as shown in Figure 4.5.

4.3.2 Physicochemical properties of jatropha and castor oil biolubricants

The result of the physicochemical characterisation of the produced jatropha biolubricant (JBL) and the castor oil biolubricant (CBL) is shown in Table 4.5. The specific gravity of CBL (0.930) is greater than the specific gravity of the JBL (0.881). Also, the density of CBL (930

kgm^{-3}) is higher than that of JBL (881 kgm^{-3}). The castor biolubricant is denser because of the presence of the hydroxyl group in it. The two biolubricants are slightly denser than the commercial mineral oil SAE20/W50 which has a specific gravity of 0.878.

Table 4.5: Physicochemical Properties of jatropha and castor oil biolubricants

S/No.	Parameter	Jatropha oil biolubricant (JBL)	Castor oil biolubricant (CBL)	SAE 20/W50
1	Specific Gravity	0.881	0.930	0.878
2	Saponification Value (mg KOH/g)	227.68	174.4	-
3	Iodine Value (gI_2/100g oil)	83.8	90.8	80
4	pH	7.19	8.16	7.12

The pH of the JBL is 7.19 and the pH of CBL is 8.16 which are both alkaline and above that of the mineral lubricant SAE 20/W50 in market which is 7.12. Therefore, the produced jatropha and castor oil biolubricants are friendly to the skin of operators that will be the end users than mineral oil based lubricants and will better protect the surfaces of machines lubricated from corrosion than SAE 20/W50. The pH of the castor oil biolubricant is higher than that of jatropha biolubricant. The biolubricants have better pH than the modified and unmodified jatropha and castor oils and will therefore have better corrosion protection on machine parts lubricated.

The saponification value of the produced jatropha biolubricant (227.6 mgKOH/g) is higher than the saponification value of the castor oil biolubricant (174.4 mgKOH/g). Both produced

biolubricants have high saponification values therefore may require blending with some additive to suppress foaming before being used to lubricate industial machines.

The iodine value of the castor biolubricant (90.8 gI_2/100g oil) is higher than the iodine value of jatropha biolubricant (83.8 gI_2/100g oil). The iodine value of both produced biolubricants is very close to that of SAE 20/W50 which is 80.090.8 gI_2/100g oil. This shows that there are still some levels of unsaturation in the biolubricants which implies that the biolubricants have a tendency to be oxidised while in service and leave some undesirable deposits on machine parts. These biolubricants may require blending with antioxidant additives to improve their performance.

4.3.3 Rheological and temperature properties of jatropha and castor oil biolubricants

The rheological properties (viscosity and viscosity index) of the produced biolubricants as well as that of mineral oil base lubricant (MNL) are depicted in Figure 4.7. From the results jatropha oil biolubricant has a kinematic viscosity of 81.7cSt at 40^0C while castor oil has kinematic viscosity of 103.2 cSt at 40^0C. The kinematic viscosity of the castor oil biolubricant is more than that of the jatropha oil biolubricant and this means that JBL has a lower lubrication capability compared to CBL. The viscosity at 100^0C of the castor oil biolubricant is also higher than that of the jatropha oil biolubricant. However, both produced biolubricants have viscosities at 40^0C and 100^0C that is less than that of petroleum oil based lubricant.

The jatropha oil biolubricant has a higher viscosity index (156.3) than the castor oil biolubricant (145.8). Therefore, the jatropha oil biolubricant will show less variation in viscosity at high temperatures compared to the castor oil biolubricant. Both biolubricants have a better viscosity index than mineral oil base lubricant SAE 20/W50.

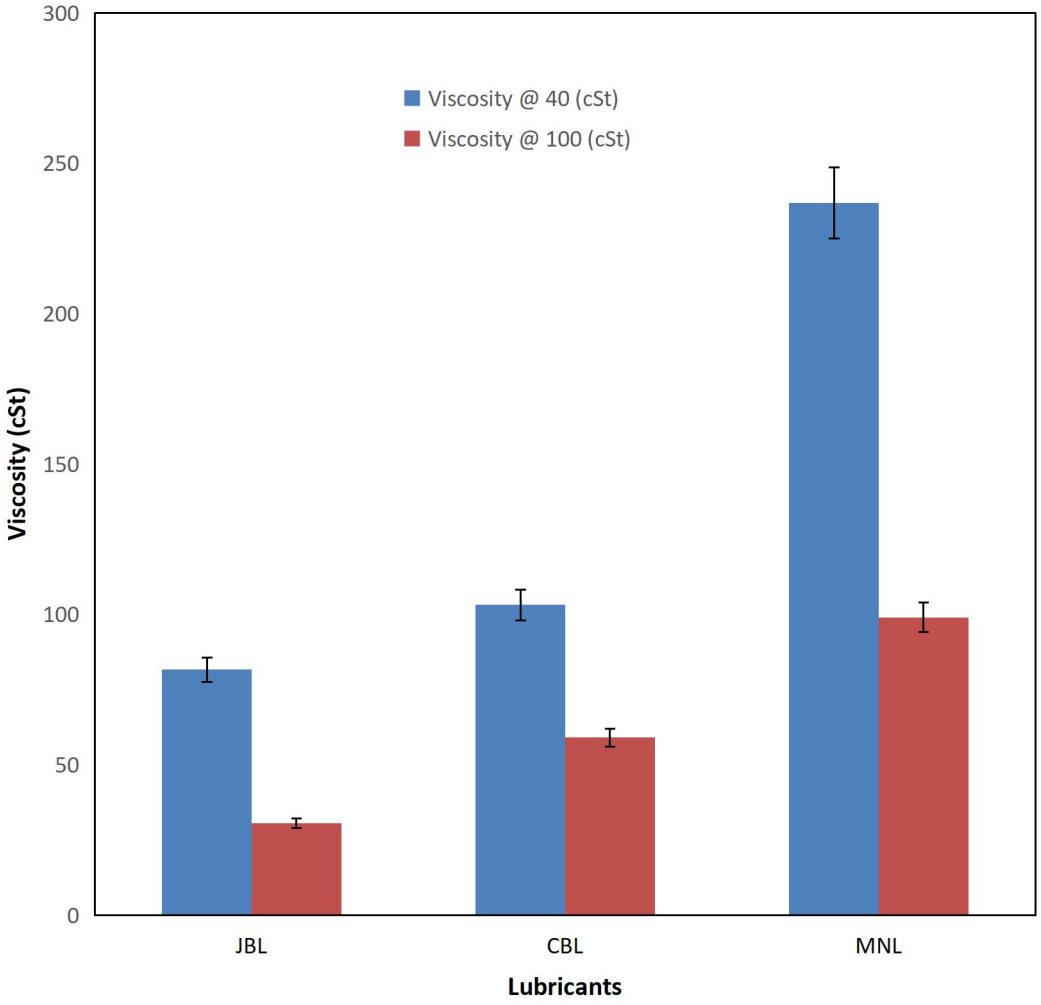


Figure 4.7: Kinematic viscosity of Jatropha and castor oil biolubricants and mineral oil lubricant

The temperature characteristics (cloud-point temperature, pour-point temperature and flash-point temperature) of the biolubricants and the mineral oil SAE 20/W50 are shown in Figure 4.8. The pour and cloud points of the castor oil biolubricant (-24.2^{0}C and -33.3^{0}C respectively) are lower than that of jatropha biolubricant (-12.^{0}C and -15.6^{0}C respectively) and that of mineral oil lubricant (-18.9^{0}C and -24.1^{0}C respectively). Thus, the castor oil biolubricant has the best cold flow property. Both JBL and CBL are suitable for low temperature applications.

The flash points of JBL and CBL are high, and this indicates that they are safe for use from the fire hazard point of view. The flame point of CBL is at par with that of mineral oil SAE 20/W50 and higher than that of JBL.

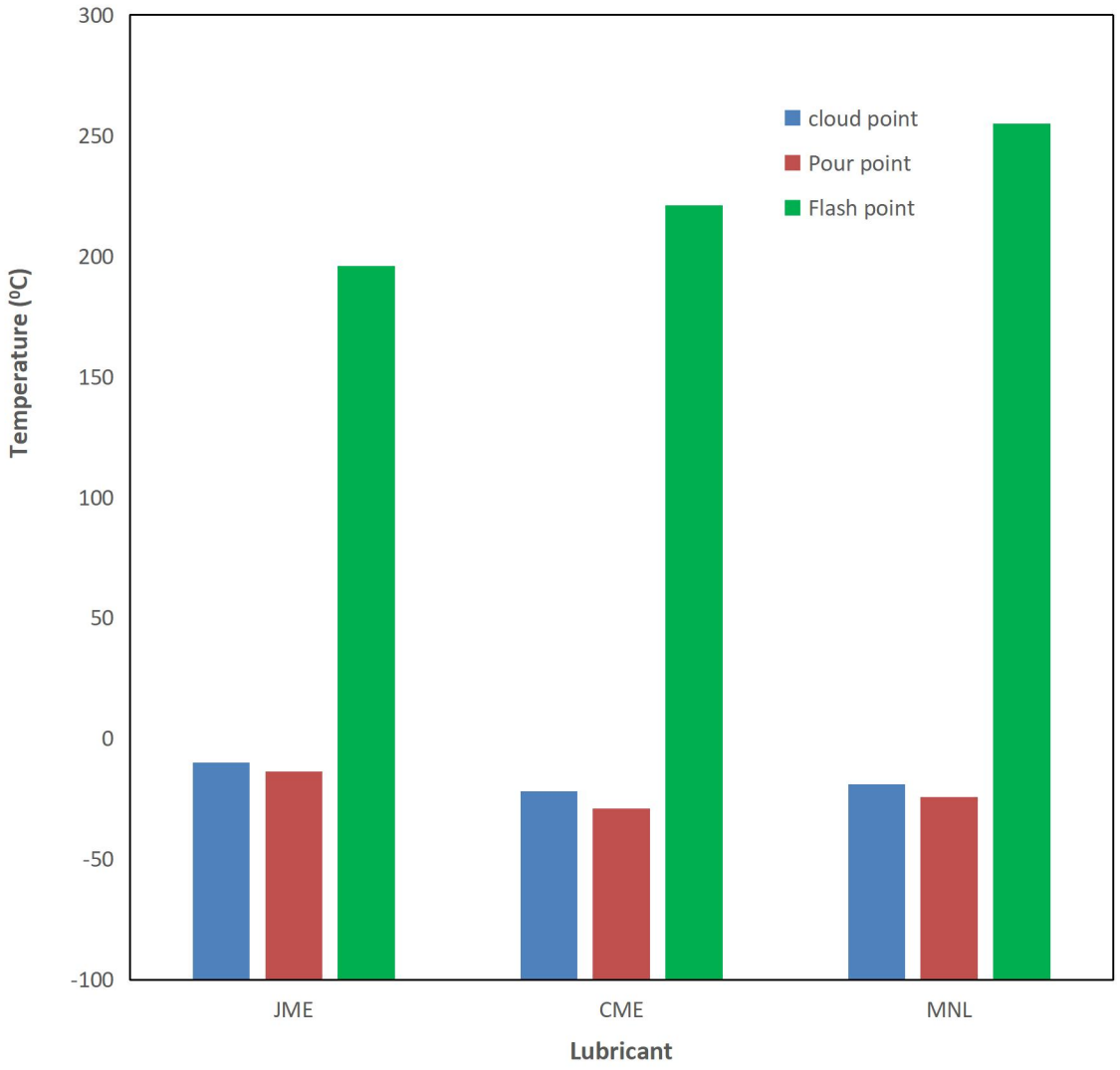


Figure 4.8: Temperature properties of Jatropha and castor oil based biolubricant and mineral oil lubricant

4.3.4 Thermo-oxidative stability of jatropha and castor oil based biolubricant

The thermo-oxidative stability of the biolubricants is determined in terms of their peroxide values. The perioxide value of the developed jatropha biolubricant (JBL) is 1.99 meq/kg while that of the developed castor biolubricant (CBL) is 2.0 meq/kg. These values are slightly

greater than that of mineral oil lubricant but much lower than that of the crude vegetable oils. The biolubricants have good thermal and oxidative stabilities but their stabilities are less than that of mineral oil SAE20/W50.

4.3.5 Corrosion inhibition characteristics of jatropha and castor oils biolubricants

Plate XIII (a) shows the humidified cast iron chippings on filter paper covered in a petri dish while Plata XIII (b) is the jatropha oil biolubricant (JBL) filter paper after 2 Hrs and Plate XIII (c) shows the castor oil biolubricant (CBL) filter paper after 2Hrs. No rust spot was found on both filter papers containing cast iron soaked in JBL and CBL. Thus, both JBL and CBL exhibits excellent corrosion inhibition characteristics and based on Alves and Oliveira (2008), both jatropha oil biolubricant and castor oil biolubricant are of corrosion grade 0.

(a)Tests with cast iron particles (b) after 2Hrs, EJO (c) after 2Hrs, ECO

Plate XIII: Corrosion inhibition characteristics of jatropha and castor oils based biolubricants

4.3.6 Biodegradability of jatropha and castor oils base lubricants

The percentage biodegradability of jatropha and castor oil-based lubricants in comparison to the commercially available petroleum-based lubricant is shown in Figure 4.9. The result of the visible ultraviolet ray spectroscopy at 520 nm wavelength is shown in Table 4.6. The castor oil had the best percentage biodegradability of 96.6% followed by modified jatropha oil with 91% biodegradability after 28 days of inoculation with bacteria. The jatropha oil, jatropha biolubricant, modified castor oil and castor oil biolubricant had the same percentage biodegradability of 81%. The commercial mineral oil-based lubricant had the lowest percentage biodegradability of 35.2%.

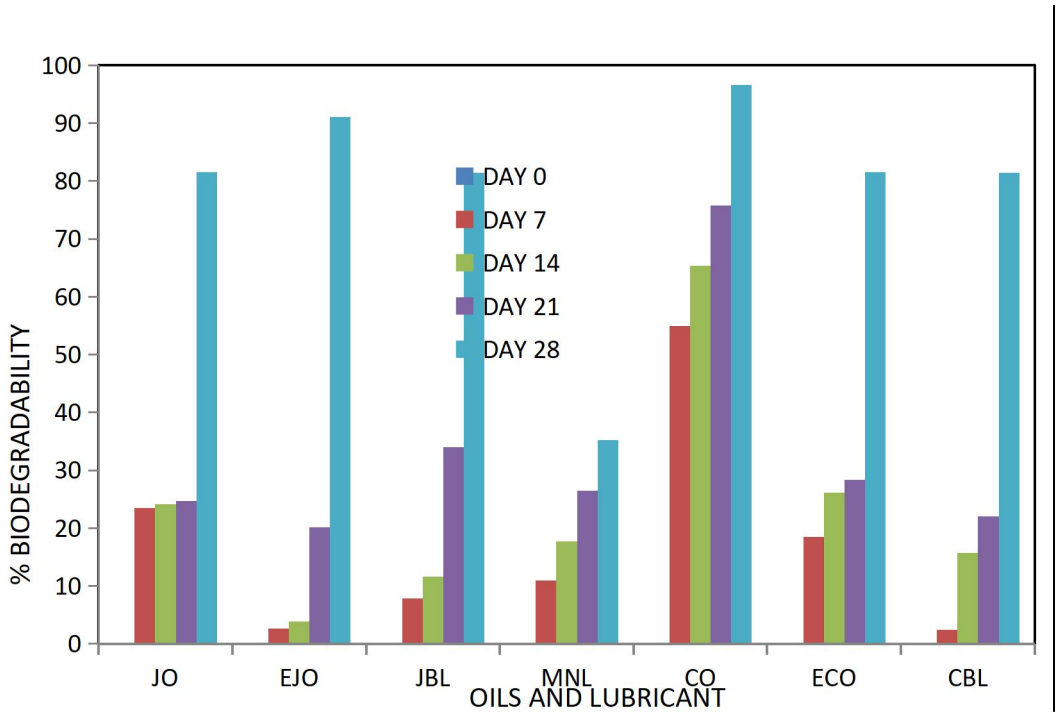


Figure 4.9: Biodegradability of jatropha and castor oils base lubricants

The jatropha and castor oil based lubricants are readily biodegradable since over 60% of the lubricants were degraded by the bacteria within 28 days. The mineral oil based lubricant is not readily biodegradable. The result is similar to results gotten with tests carried out according to the Coordinating European Council biodegradability test method (Aluyor *et al;* 2009). The modification of jatropha oil increased its biodegradability, while the modification

of castor oil reduced its biodegradability. The developed jatropha and castor biolubricants had the same biodegradability.

Table 4.6: Visible ultraviolet ray spectroscopy absorbance in nanometer (nm) of jatropha and castor oils base lubricants

Oils/Lubricant	Absorbance (nm) after Days				
	0	7	14	21	28
JO	1.999	1.997	1.491	0.919	0.297
EJO	1.375	1.042	1.023	0.698	0.053
JBL	1.336	0.776	0.716	0.465	0.111
MNL	1.679	1.127	0.668	0.653	0.583
CO	0.694	0.676	0.458	0.452	0.434
ECO	1.345	0.805	0.778	0.483	0.370
CBL	1.342	1.132	0.938	0.862	0.175

Similarly, from the Table 4.6, the mineral oil had the highest absorbance of 0.583 nm after 28 days of inoculation with bacteria while the modified jatropha oil had the lest absorbance of 0.053 nm. The visible U-V spectroscopy result confirms that the jatropha and castor oils-based lubes are more biodegradable compared to mineral oil base lubricant. The jatropha and castor oil based lubricants were readily consumed by the bacteria such that their solution became less turbid and absorbed less U-V rays. The mineral oil based lubricant that was not readily consumed had a more turbid solution thereby absorbing more U-V rays.

4.4 Tribological Properties of Jatropha and Castor Oil Base Lubricants

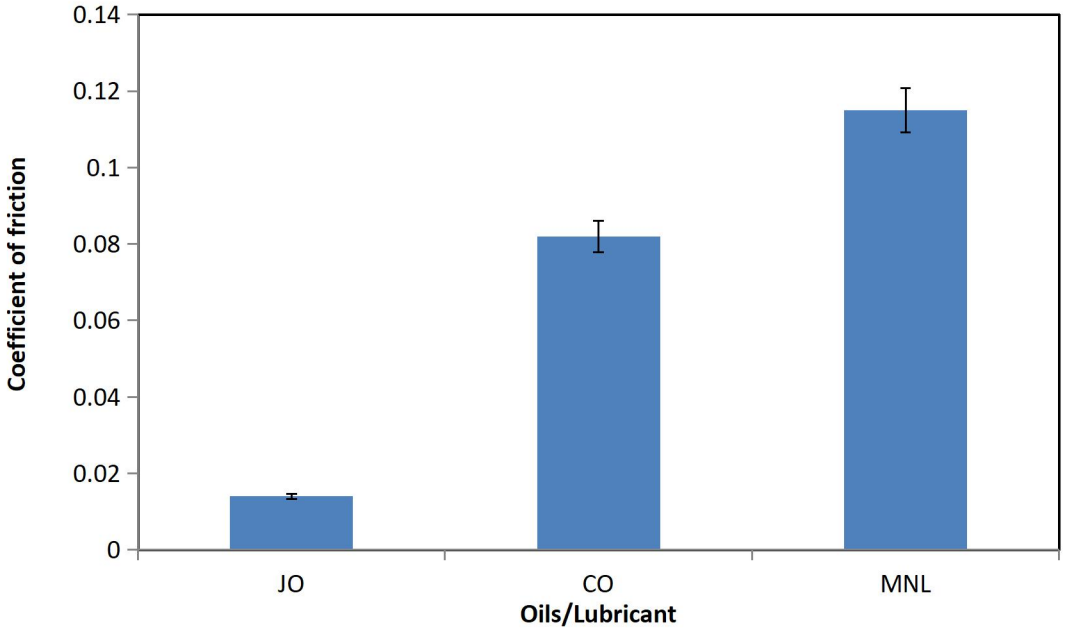
The basic duty of any fluid lubricant includes limiting wear, friction and damage to surfaces for the entire designed life-time of a mechanical system. Tribological evaluation of fluid lubricants involves the measurement of frictional forces, friction coefficient, wear rate and wear scar diameter. The following subsections presents and discusses the results obtained from the ball on disc standard tribometer measurements carried out on the jatropha and castor oils based lubricants.

4.4.1 Tribological properties of jatropha and castor oils

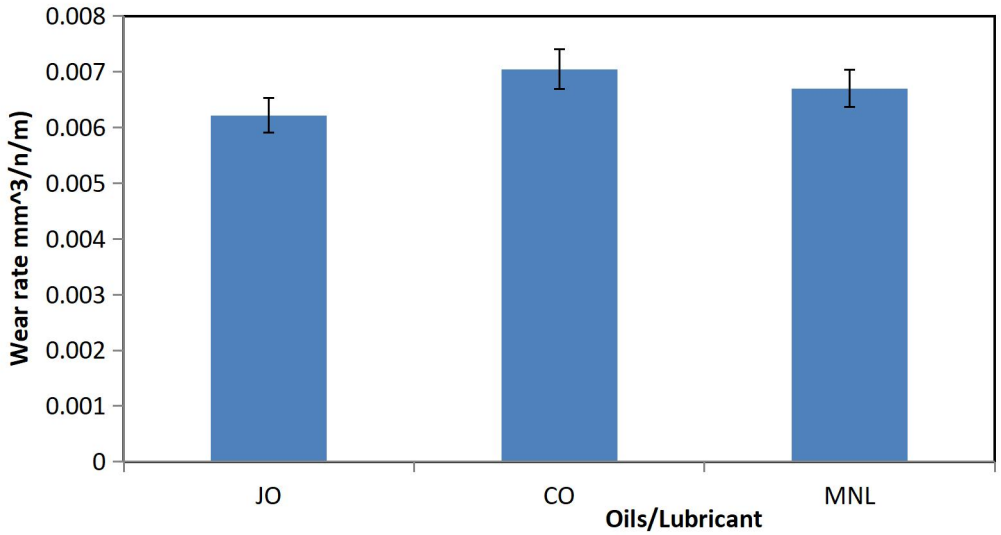
The coefficient of friction of the jatropha and castor oil vis-a-vis to that of commercial mineral oil is shown in Figure 4.10 (a) while the wear rate is shown in Figure 4.10 (b).

Both the jatropha and castor oils had lower coefficient of friction (0.014 and 0.082 respectively) compared to the commercial mineral oil based lubricant which had coefficient of friction of 0.115. This means that the jatropha and castor oils had better friction reduction ability than SAE 20/W50. Thus, a machine using these vegetable oils as lubricant will have less energy loss due to friction compared to the machine using SAE 20/W50.Thus, the fuel consumption of a machine lubricated with jatropha or castor oil will be lower than that of a machine lubricated with SAE 20/W50. This result is in concord with the findings of earlier researchers (Ratoi *et al*., 2000., Weijiu *et al*., 2003., Shahabuddin *et al*; 2013a; Shahabuddin *et al*; 2013b., Golshokouh *et al*; 2014; Binfa *et al*., 2015).

The jatropha and castor oil did better than the mineral oil based lubricant in friction due to their ester functionality. The positively charged heads of the fatty acid chains adsorbed to the metal surfaces by a chemical reaction that allowed a monolayer film formation; while the negative end of the fatty acids (hydrocarbon chains) stocked away almost perpendicularly from the metal surface. This ester functionality (property) of vegetable oils is called "lubricity" (Ratoi *et al*., 2000). The fatty acid single bond ($-CH_2-$) chain offered a sliding space that prevented the two metal surfaces from making body contact with each other. Additionally, the fatty acids in the vegetable oils reacted with the metals to form metallic salts that absorbed to the metal surfaces in contact to form a tribo-film. The metallic salt tribo-film is of low shear which further reduced the friction. Adhvaryu and Erhan (2002), gave detail explanation of the chemical reaction taking place between the vegetable oil and the metals at the contact surface.



(a) Friction coefficient of jatropha and castor oils



(b): Wear rate of jatropha and castor oils

Figure 4.10: Friction coefficient and wear rate of jatropha, castor oils and mineral based lubricant (a) Friction coefficient(b) Wear rate

The coefficient of friction of the jatropha oil (0.014) is less than the friction coefficient of the castor oil (0.082). This lower coefficient of friction of jatropha oil may be because the oil had better lubricity than the castor oil. Also, it might be that jatropha oil tribo-film is of a lower shear strength than the castor oil tribo-film therefore the jatropha oil gave a lower friction coefficient. The friction coefficient obtained while using jatropha oil as lubricant is lower than the result reported by Mushtaq and Hanief (2021), this is because of the difference in materials used to carry out the tests and the slight change in methods used. While this research used steel ball on aluminium disc, Mushtaq and Hanief (2021) used steel pin on steel disc.

From the result in Figure 4.10b jatropha oil had the least wear rate of 0.0062 $mm^3N^{-1}m^{-1}$ followed by the mineral oil with wear rate of 0.0067 $mm^3N^{-1}m^{-1}$ while the castor oil had the highest wear rate of 0.007 $mm^3N^{-1}m^{-1}$. The castor oil competed favourably with the SAE 20/W50 while the jatropha oil exceeded SAE 20/W50 in wear protection performance. This result is the same as the findings of Asadauskas *et al*., (1996) and Stojilkovic and Kolb (2016). Also, Binfa *et al*., (2015) found that the wear performance of mineral oil based lubricant exceeded that of castor oil because of the presence of strong antiwear additives in the mineral base lubricant.

The jatropha oil performed better in wear protection than the SAE 20/W50 lubricant contrary to the findings of most researchers (Ajithkumar, 2009., Syahrullail *et al.* 2013 and Shahabuddin *et al*., 2013b) that reported vegetable oils as performing less than commercial mineral oil base lubricants. The poor performance in wear protection of the SAE 20/W50 compared to the jatropha oil indicates that the additives in the SAE 20/W50 are counter-productive as they compete with each other for the surface of the metals. The better performance of the jatropher oil over the castor oil confirms the earlier finds of Syahrullail *et al*., (2013) where it was discovered that jatropha oil had better wear protection performance

than palm oil and palm fatty acid distillates. It is concluded that jatropha oil has better tribological properties than castor oil and SAE 20/50.

4.4.2 Tribological properties of modified jatropha and castor oils

The friction coefficient of the modified jatropha and castor oils as compared with mineral base oil and unmodified jatropha and castor oils is as displayed in Figure 4.11. The corresponding rate of wear is shown in Figure 4.12.

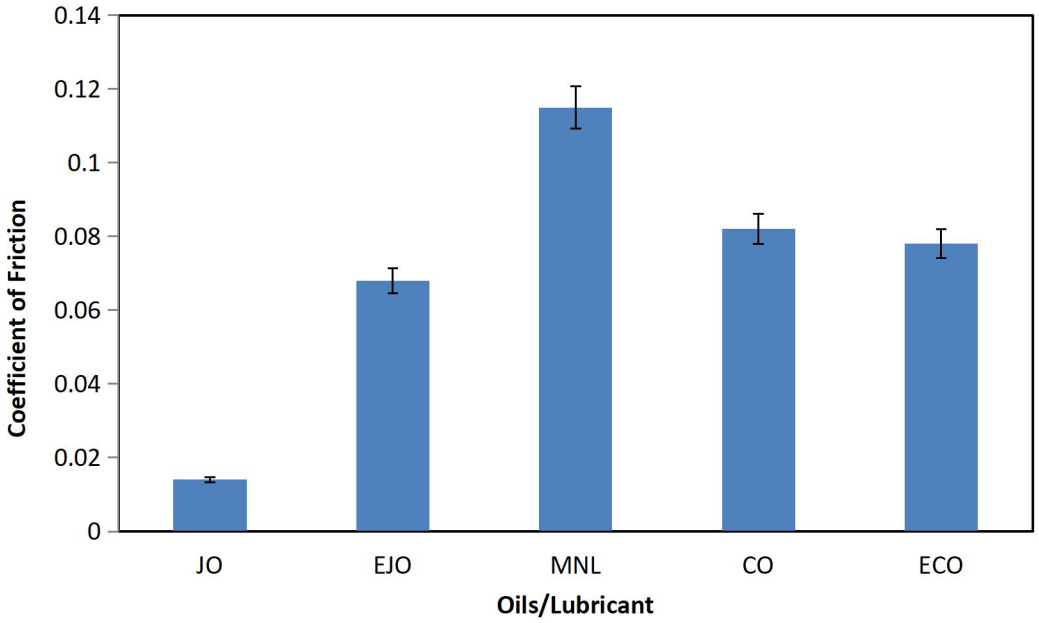


Figure 4.11: Friction coefficient of modified jatropha and castor oils

The friction coefficient of the modified jatropha oil (0.068) is less than that of the mineral oil base lubricant (0.115). Also, the friction coefficient of the modified castor oil (0.078) is less compared to petroleum-based lubricant. Modified jatropha oil performed better than the modified castor oil in friction reduction.

The friction coefficient of the modified jatropha oil was much higher than the one of unmodified jatropha oil. Conversely, the friction coefficient of the modified castor oil was less than that of the unmodified castor oil. It can be concluded that both modified jatropha and castor oils have better friction reduction properties than the commercial mineral oil base

lubricant (SAE 20/W50). Also, the modification of jatropha oil through acid catalysed esterification reduces its friction protection performance. The modification of castor oil through acid catalysed esterification improves its friction reduction properties.

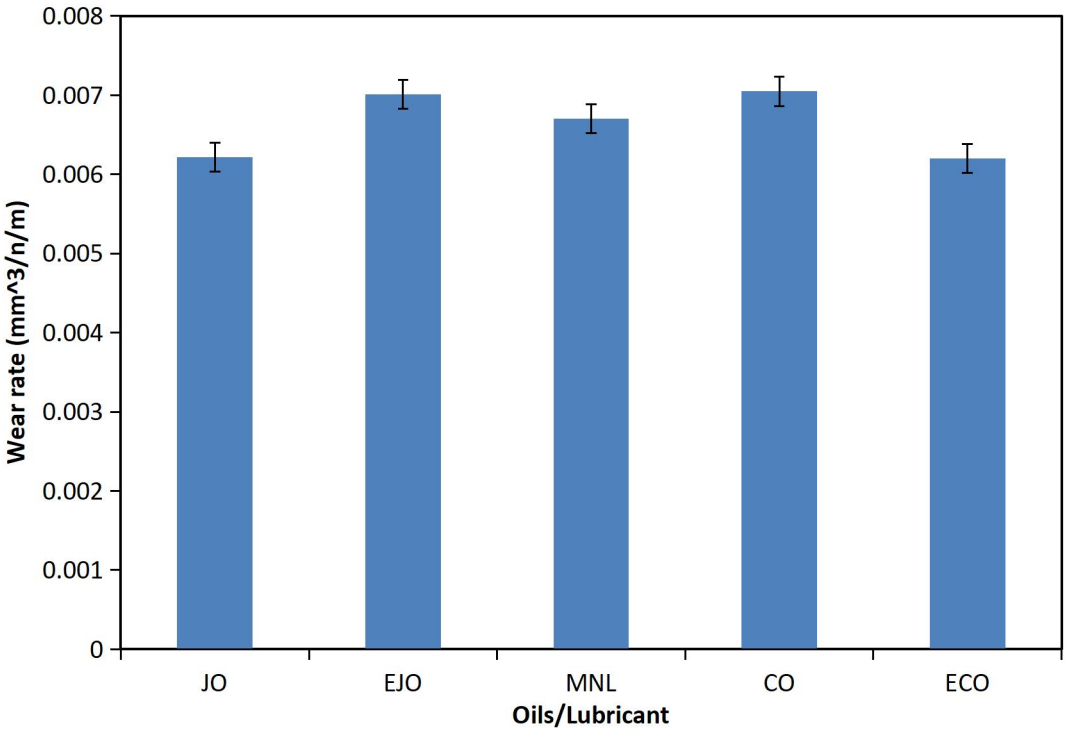


Figure 4.12: Wear rate of modified jatropha, castor oils and mineral based lubricant

The wear rate of the modified jatropha oil (0.007008 $mm^3N^{-1}m^{-1}$) was higher than the wear rate of the mineral oil base lubricant (0.006702 $mm^3N^{-1}m^{-1}$). The rate of wear of the modified castor oil (0.0062 $mm^3N^{-1}m^{-1}$) was lower than that of the mineral oil. The rate of wear of the modified castor oil was much less than that of modified jatropha oil. The wear rate of the modified jatropha oil is much higher than that of the unmodified jatropha oil. The wear rate of the modified oil from castor bean is much less compared with unmodified castor oil.

The modified oil from castor bean has better wear protection behaviour than the commercial petroleum oil based lubricant (SAE 20/W50). The mineral oil has better wear performance than the modified jatropha oil. It is concluded that modification of jatropha oil through acid

catalysed esterification deteriorates its tribological properties whereas the same modification of castor oil improves its tribological properties. Modified castor bean oil exhibited better tribological behaviour than the commercial petroleum oil base lubricant (SAE 20/W50). Modified jatropha oil performs better in friction reduction than the mineral oil base lubricant but has poorer wear protection than the mineral oil base lubricant SAE 20/W50).

4.4.3 Tribological character of the developed jatropha and castor bean oil biolubricants

The coefficient of friction of the developed jatropha and castor oil biolubricants as compared to the mineral oil base lubricant and other jatropha and castor oil base lubricants is shown in Figure 4.13. The chart of the wear rate of the developed jatropha and castor oil biolubricants is as shown in Figure 4.14. Both the jatropha and castor oil biolubricants had lower friction coefficient (0.075 and 0.067 respectively) compared to mineral base lubricant (0.115). Jatropha biolubricant Friction coefficient is larger than the friction coefficient of both modified and unmodified jatropha oil. Whereas the friction coefficient of developed castor biolubricant is less compared to both the modified and unmodified castor oils. The vegetable (jatropha and castor) oil based lubricants had better friction reduction properties than the commercial mineral oil based lubricant (SAE 20/W50).

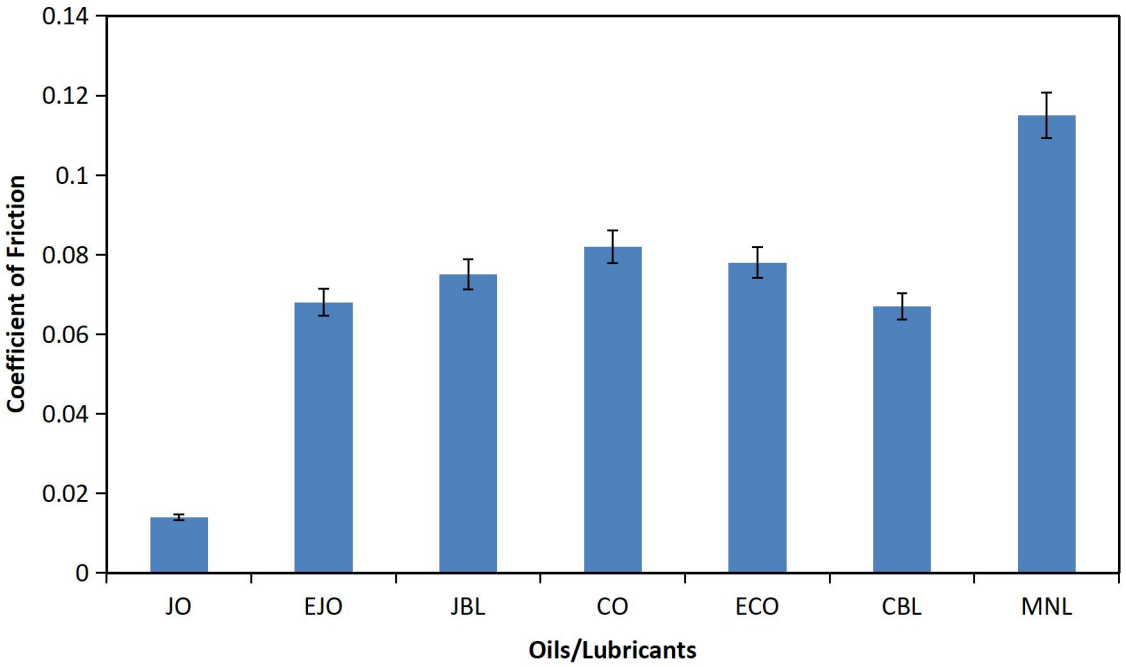


Figure 4.13: Friction coefficient of the developed jatropha, castor biolubricants and mineral based lubricant

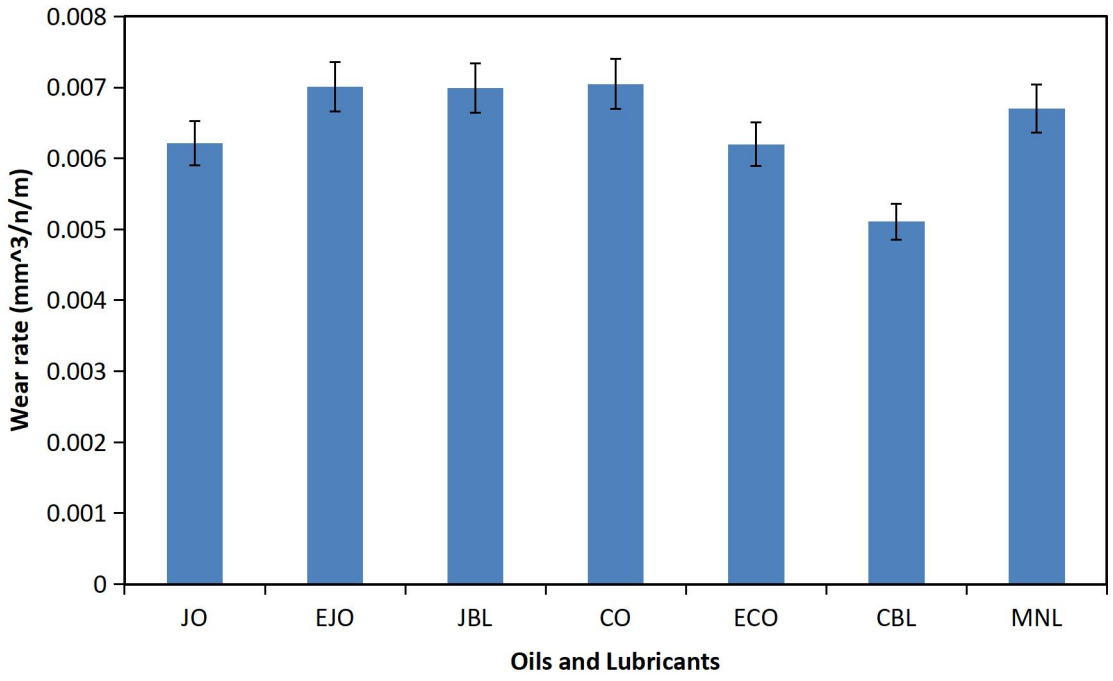


Figure 4.14: Wear rate of developed jatropha, castor biolubricants and mineral based lubricant

The wear rate of the developed jatropha oil biolubricant was 0.00699 $mm^3N^{-1}m^{-1}$which is higher than the wear rate of the mineral base oil (0.00670 $mm^3N^{-1}m^{-1}$). The castor biolubricant wear rate (0.00511 $mm^3N^{-1}m^{-1}$) is less than that of the mineral oil base lubricant. The wear rate of the jatropha biolubricant is the same as that of modified jatropha oil while both are higher than the rate of wear of jatropha oil. The rate of wear of the developed castor oil biolubricant is lower than the wear rate of both castor oil and modified castor oil.

The developed castor bean oil biolubricant tribological characteristics exceeded that of jatropha biolubricant and petroleum oil based lubricant. The developed jatropha biolubricant performed better in friction but less in wear protection than the commercial mineral oil base lubricant (SAE 20/W50). It can be deduced that transesterification of the jatropha oil with TMP polyol deteriorates its tribological properties whereas transesterification of castor oil with TMP polyol improves its tribological properties.

4.5 Additives Influence on the Tribological Performance of Oils and Lubricants Developed

The experimental process parameters and the corresponding responses are shown in Tables 4.7 and 4.8.

Table 4.7: Experimental Process Parameters and Responses for oils

Run order	Experimental design				Jatropha oil			Castor oil		
	A	B	C	D	Coef. Friction	Wear rate(10^-3 mm^3/n/m)	VI	Coef. Friction	Wear rate(10^-3 mm^3/n/m)	VI
1	1	1	0.3	2	0.065	5.419	185.5733	0.032	5.213	167.9414
2	1	2	0.6	2.5	0.076	5.638	183.0165	0.084	6.018	167.93
3	1	3	0.9	3	0.074	6.303	184.4925	0.086	5.157	163.7804
4	2	1	0.6	3	0.071	6.964	186.7008	0.082	5.271	164.3125
5	2	2	0.9	2	0.075	6.727	183.3441	0.086	5.637	167.2565
6	2	3	0.3	2.5	0.071	6.051	193.8307	0.092	5.547	163.6433
7	3	1	0.9	2.5	0.074	0.0001	198.0171	0.088	5.58	168.2057
8	3	2	0.3	3	0.082	6.622	202.721	0.093	5.663	166.9373
9	3	3	0.6	2	0.068	3.881	212.2114	0.093	5.074	166.122

Table 4.8: Experimental Process Parameters and Responses for Biolubricants

Run order	Experimental design				Castor Biolubricant			Jatropha Biolubricant		
	A	B	C	D	Coef. Friction	Wear rate (10^-3 mm^3/n/m)	VI	Coef. Friction	Wear rate (10^-3 mm^3/n/m)	VI
1	1	1	0.3	2	0.088	6.453	180.580	0.124	4.913	196.923
2	1	2	0.6	2.5	0.094	5.502	179.734	0.114	4.83	231.225
3	1	3	0.9	3	0.087	4.324	178.255	0.073	3.864	227.961
4	2	1	0.6	3	0.084	5.006	192.679	0.097	4.375	184.546
5	2	2	0.9	2	0.068	3.546	173.068	0.042	4.188	180.135
6	2	3	0.3	2.5	0.081	5.082	191.961	0.086	3.364	179.728
7	3	1	0.9	2.5	0.069	7.407	202.626	0.079	3.661	182.408
8	3	2	0.3	3	0.126	7.473	192.988	0.078	3.851	178.791
9	3	3	0.6	2	0.082	6.627	220.769	0.084	4.829	180.918

4.5.1 Effect of additives on the coefficient of friction of oils and biolubricants developed

The normal probability plot displaying the significant additives affecting the friction coefficient for each oil and lubricant is shown in Figure 4.15. The viscosity modifier additive (B) had the dominant influence on the friction coefficient of the jatropha oil. The anti-wear additive (A) had the dominant influence on the friction coefficient of the castor oil.

The extreme pressure additive (c) had the dominant influence on the friction coefficient of the castor and jatropha biolubricants. None of the combination of any of the two additives became more significant in influencing the coefficient of friction. The percentage concentration of the extreme pressure additive (c) has the dominant influence on the friction coefficient of developed biolubricants.

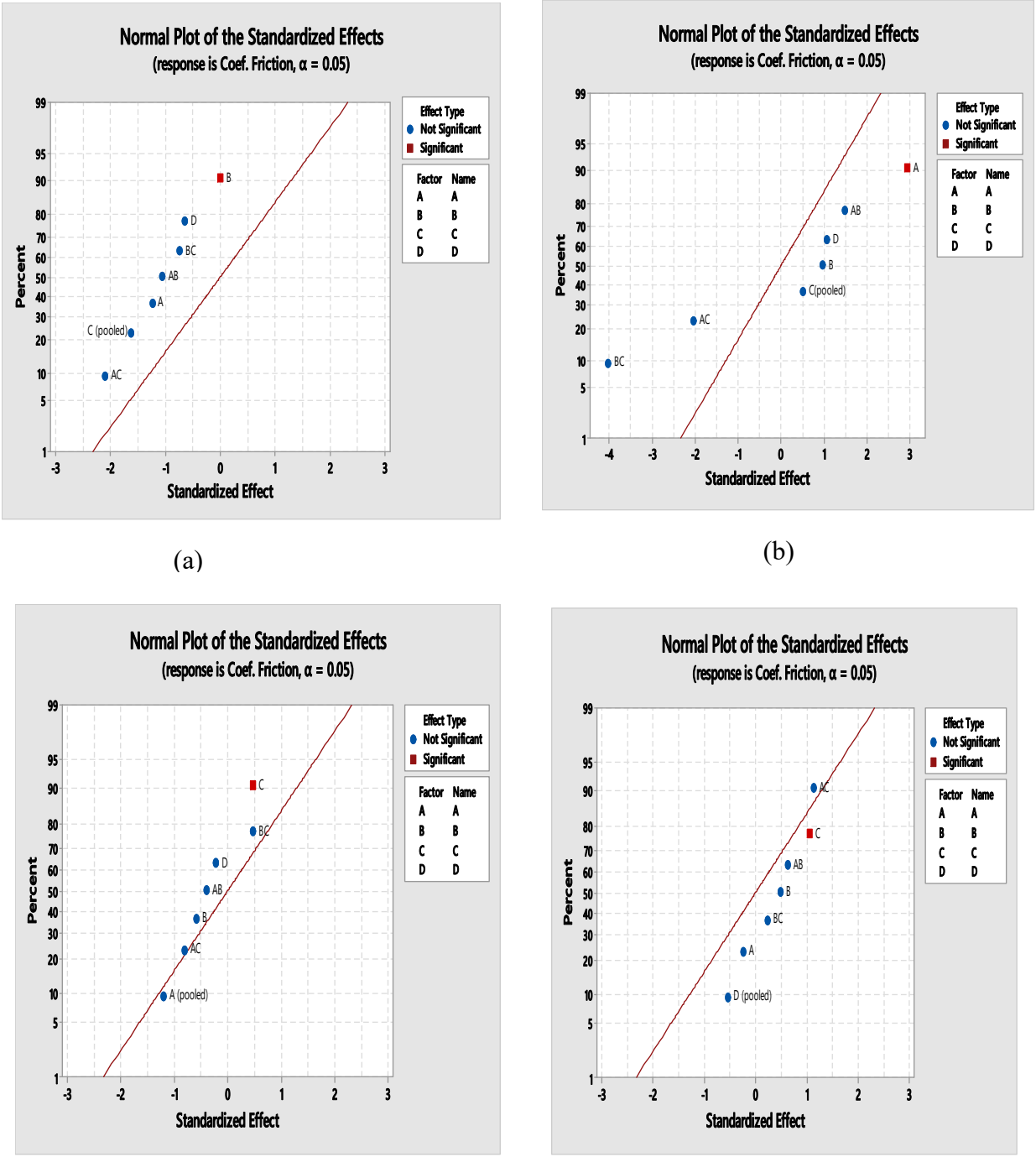


Figure 4.15: Normal Probability Plot of Coefficient of Friction (a) Jatropha oil (b) Castor oil (c) Castor biolubricant (d) Jatropha biolubricant

4.5.1.1 Analysis of variance (ANOVA) of coefficient of friction of oils and biolubricants developed

(a) Jatropha oil

The ANOVA for the coefficient of friction of the jatropha oil is shown in Table 4.9a. The viscosity modifier additive (B) with 53.89% contribution, had the dominant influence on the coefficient of friction. The anti-corrosion additive (D) with 32.64% contribution, had the second dominant influence on the friction coefficient of the jatropha oil. Anti-wear additive A (7.77%) had the least significance and C was a contribution to the error. The error contributed 5.7% to the coefficient of friction.

Table 4.9a: ANOVA for Coefficient of Friction of Jatropha oil

Factor	**DOF**	**SS**	**MS**	**F**	**P (%)**
A	2	0.000015	7.5E-06	1.3636	7.77
B	2	0.000104	0.000052	9.455	53.89
C	poooled	pooled	pooled	pooled	Pooled
D	2	0.000063	3.15E-05	5.727	32.64
Pooled Error	2	0.0000110	5.5E-06		5.70
Total	8	0.000193	2.41E-05		100.00

(b) Castor oil

The ANOVA for the coefficient of friction of castor oil is shown in Table 4.9b. The anti-wear additive A (33.36 %) had the dominant influence on the coefficient of friction, followed by the viscosity modifier additive B (32.60 %). The anti-corrosion additive D (20.28 %) had the least significance and the extreme pressure additive C was a contribution to the error. there was a percentage error of 13.76, the error is this high because the extreme pressure additive C contributed to the error.

Table 4.9b: ANOVA for Coefficient of Friction of Castor oil

Factor	DOF	SS	MS	F	P (%)
A	2	0.000972	0.000486	2.4239	33.36
B	2	0.000950	0.000475	2.369	32.60
C	pooled	pooled	pooled	Pooled	pooled
D	2	0.000591	0.000296	1.474	20.28
Pooled Error	2	0.0004010	0.000201		13.76
Total	8	0.002914	0.000364		100.00

(c) Castor oil based biolubricant

The ANOVA for the coefficient of friction of castor biolubricant is shown in Table 4.9c. Similarly, for the castor biolubricant, the extreme pressure additive C (36.14%) had the dominant influence on the friction coefficient, followed by the anti-corrosion additive D (30.25%). The viscosity modifier additive B (20.28%) had the least significance and the anti-wear additive A was a contribution to the error. There was a 15.75%error contribution to the coefficient of friction.

Table 4.9c: ANOVA for Coefficient of Friction of Castor oil based Biolubricant

Factor	DOF	SS	MS	F	P
A	pooled	pooled	pooled	Pooled	pooled
B	2	0.000415	0.000208	1.134	17.86
C	2	0.00084	0.00042	2.295	36.14
D	2	0.000703	0.000352	1.921	30.25
Pooled Error	2	0.0003660	0.000183		15.75
Total	8	0.002324	0.000291		100.00

(d) Jatropha oil based biolubricant

The ANOVA for the coefficient of friction of jatropha biolubricant is shown in Table 4.9d. The extreme pressure additive C with 46.41% contribution had the dominant influence on the friction coefficient, followed by the anti-wear additive A (30.53%). The viscosity modifier additive B (18.69%) had the least significance and the anti-corrosion additive D was a contribution to the error. There was a 4.38%error contribution to the coefficient of friction.

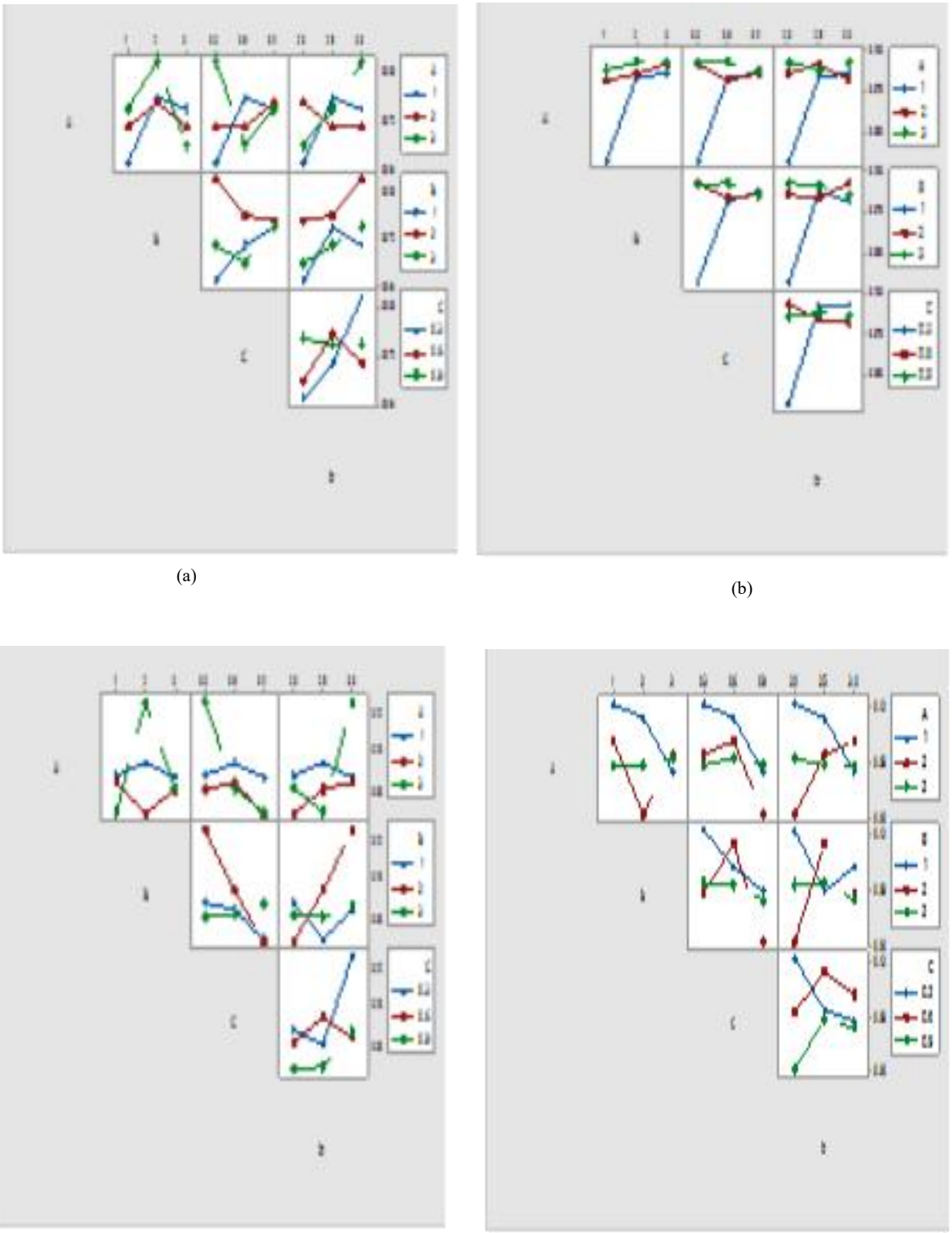
Table 4.9d: ANOVA for Coefficient of Friction of Jatropha oil based Biolubricant

Factor	DOF	SS	MS	F	P
A	2	0.001395	0.000698	6.9750	30.53
B	2	0.000854	0.000427	4.270	18.69
C	2	0.002121	0.001061	10.605	46.41
D	pooled	pooled	pooled	pooled	Pooled
Pooled Error	2	0.0002000	1E-04		4.38
Total	8	0.004570	0.000571		100.00

4.5.1.2 Interactions influence on the friction coefficient of oils and lubricants developed

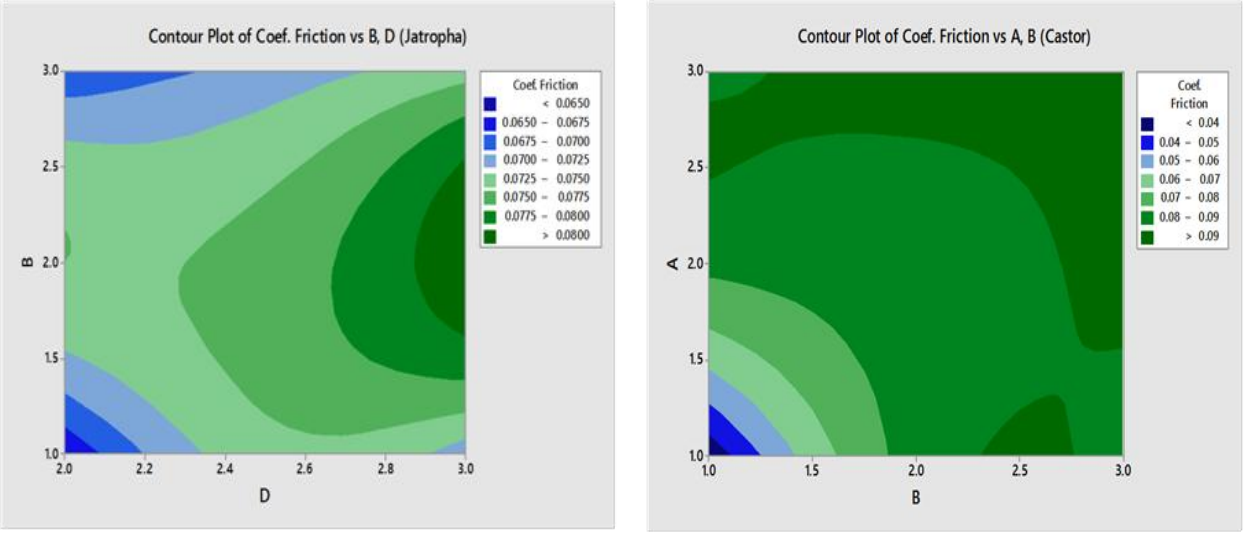
The interaction plot of the parameters for friction coefficient for the jatropha and castor oil based lubricants is shown in Figure 4.16. It shows that there is interaction between the four additives at all levels for the jatropha and castor oil base lubricants. This is in agreement with the findings of earlier researchers that additives interact with each other and some of the interactions can be either destructive or enhancing (Ratoi *et al*., 2000; Menezes *et al.,* 2013).

The contour plot of the coefficient of friction for the jatropha and castor oil base lubricants is shown in Figure 4.17. For the jatropha oil, as the concentration of D and B increases, the coefficient of friction increases which is not desirable. To keep friction below 0.07, B should not exceed 1.5% and D should not exceed 2.3%. For the castor oil as the concentration of A and B increases, the friction coefficient also increases. For the castor biolubricant, as the concentration of C increases, the friction coefficient reduces while as the concentration of D increases, the friction coefficient increased. For the jatropha biolubricant, as the concentration of A and C increases, the coefficient of friction decreased.

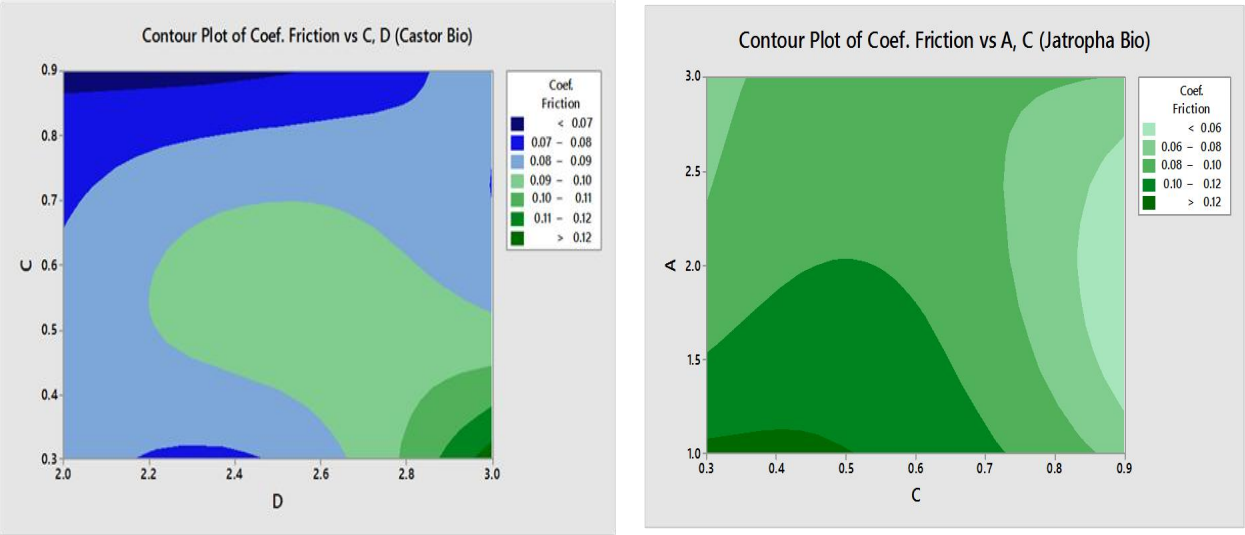


(a) (b) (c) (d)

Figure 4.16: Interaction Plot of Coefficient of Friction (a) Jatropha oil (b) Castor oil (c) Castor oil based biolubricant (d) Jatropha oil based biolubricant



(a) (b)



(c) (d)

Figure 4.17: Contour Plots of Coefficient of Friction (a) Jatropha oil (b) Castor oil (c) Castor oil based biolubricant (d) Jatropha oil based biolubricant

4.5.2 Influence of additives on the rate of wear of jatropha and castor oil based lubricants

The normal probability plot displaying the significant additives affecting the wear rate for each oil and lubricant is shown in Figure 4.18. The anti-wear additive (A) had the dominant influence on the rate of wear of the jatropha oil. The viscosity modifier additive (B) had the most significant effect on specific rate of wear of the castor oil.

The anti-wear additive (A) had the dominant impact on rate of wear of the castor biolubricant. Extreme pressure additive (c) had the most significant influence on rate of wear of the jatropha biolubricant. None of the combination of any of the two additives became more significant in influencing the wear rate.

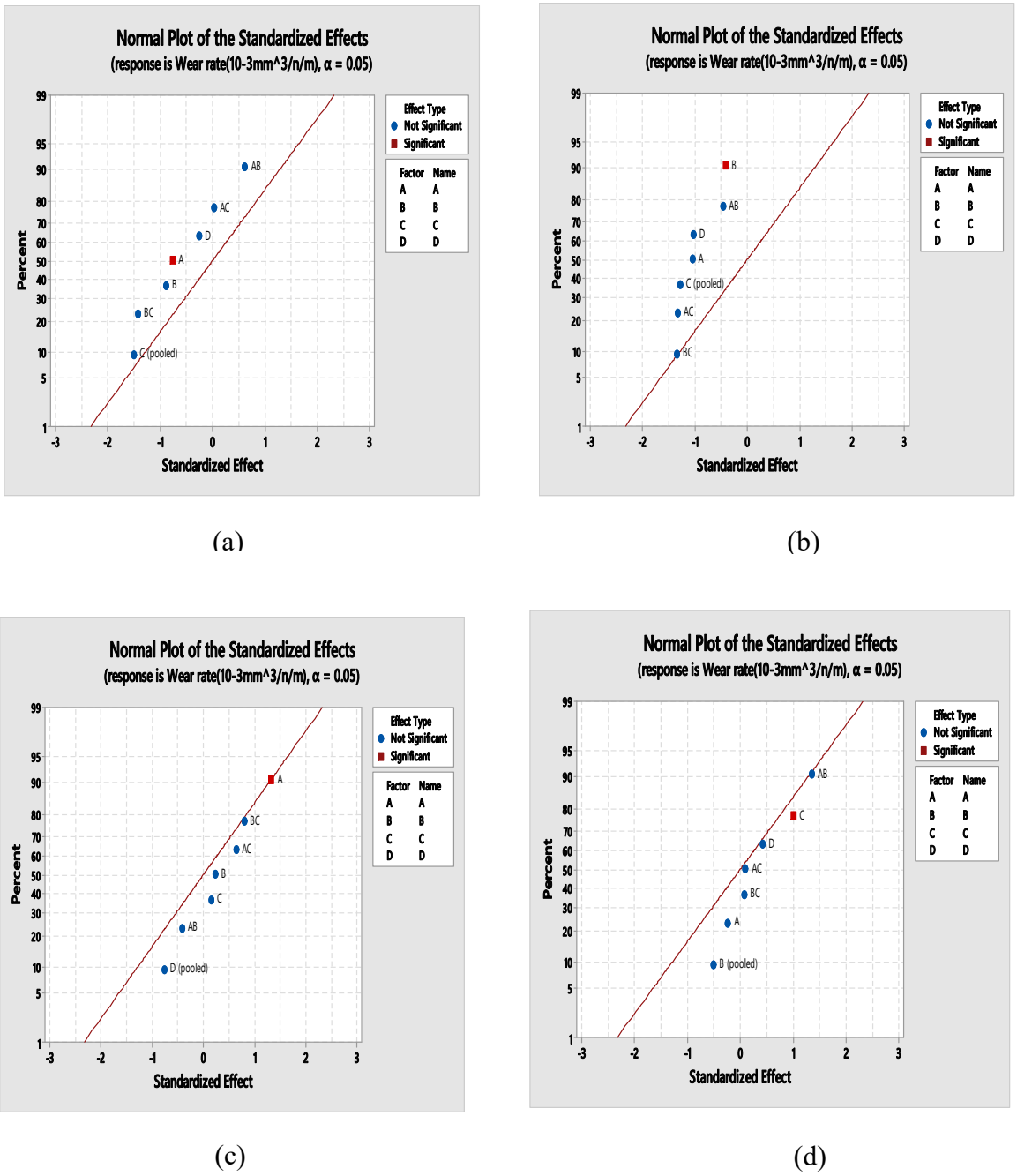


Figure 4.18: Normal Probability Plot of Wear Rate (a) Jatropha oil (b) Castor oil (c) Castor biolubricant (d) Jatropha biolubricant

4.5.2.1 Analysis of variance (ANOVA) of wear rate of oils and lubricants developed

(a) Jatropha oil

The ANOVA for the wear rate of Jatropha seed oil is shown in Table 4.10a. The most significant factor for the wear rate is the anti-wear additive A (40%) followed by the anti-corrosion additive D (29.26%). the least significant factor is the viscosity modifier additive B (19.13%) while the extreme pressure additive C contributed to the error. There is 11.61% error, the error is this large because additive extreme pressure additive C contributed to it.

Table 4.10a: ANOVA for Wear Rate of Jatropha oil

Factor	DOF	SS	MS	F	P
A	2	15.34	7.67	3.4441	40.00
B	2	7.336000	3.668	1.647	19.13
C	Pooled	Pooled	Pooled	Pooled	Pooled
D	2	11.220000	5.61	2.519	29.26
Pooled Error	2	4.4540000	2.227		11.61
Total	8	38.350000	4.79375		100.00

(b) Castor oil

The ANOVA for the wear rate of the castor oil is shown in Table 4.10b. The most significant factor is the viscosity modifier B (60.17%) followed by the anti-corrosion additive D (39.31 %). The least significant factor was the anti-wear additive A (0.43%) and the extreme pressure additive C was a contribution to the error. The percentage error for the castor oil wear rate is 0.09.

Table 4.10b: ANOVA for Wear Rate of Castor oil

Factor	DOF	SS	MS	F	P
A	2	0.003175	0.001588	4.6215	0.43
B	2	0.447300	0.22365	651.092	60.17
C	pooled	Pooled	Pooled	Pooled	pooled
D	2	0.292200	0.1461	425.328	39.31
Pooled Error	2	0.0006870	0.000343		0.09
Total	8	0.743362	0.09292		100.00

(c) Castor oil based biolubricant

The ANOVA for the wear rate of the castor oil based biolubricant is shown in Table 4.10c. The most significant contribution to rate of wear was from the anti-wear additive A (71.73%) followed by the extreme pressure additive C (15.55 %). The least significant factor was viscosity modifier additive B (10.25 %) while the anti-corrosion additive D was a contribution to the error. The error contributed 2.47 % to the wear rate of the castor biolubricant.

Table 4.10c: ANOVA for Wear Rate of Castor oil based biolubricant

Factor	**DOF**	**SS**	**MS**	**F**	**P**
A	2	10.701	5.3505	29.0788	71.73
B	2	1.529000	0.7645	4.155	10.25
C	2	2.320000	1.16	6.304	15.55
D	pooled	pooled	pooled	pooled	pooled
Pooled Error	2	0.3680000	0.184		2.47
Total	8	14.918000	1.86475		100.00

(d) Jatropha oil based biolubricant

Jatropha oil based biolubricant ANOVA for wear rate is shown in Table 4.10d. Extreme pressure additive C (39.96%) was the most significant factor followed by the anti-corrosion additive D (33.68%). The least significant factor was the anti-wear additive A (19.99 %) while viscosity modifier additive B contributed to the error. The contribution of error to the wear rate of the jatropha oil based biolubricant was 6.37%

Table 4.10d: ANOVA for Wear Rate of Jatropha oil based biolubricant

Factor	**DOF**	**SS**	**MS**	**F**	**P**
A	2	0.5107	0.25535	3.1370	19.99
B	pooled	pooled	pooled	pooled	pooled
C	2	1.021000	0.5105	6.271	39.96
D	2	0.860700	0.43035	5.287	33.68
Pooled Error	2	0.1628000	0.0814		6.37
Total	8	2.555200	0.3194		100.00

4.5.2.2 Interactions effects on wear rate of oils and biolubricants developed

The interaction plot for the four parameters (percentage additives concentration) in wear is shown in Figure 4.19. The plots shows that there was an interaction between the four additives A, B, C and D at all levels. This is in agreement with the findings of earlier researchers that additives interact with each other and some of the interactions can be either destructive or enhancing (Ratoi *et al*., 2000; Menezes *et al.,* 2013).

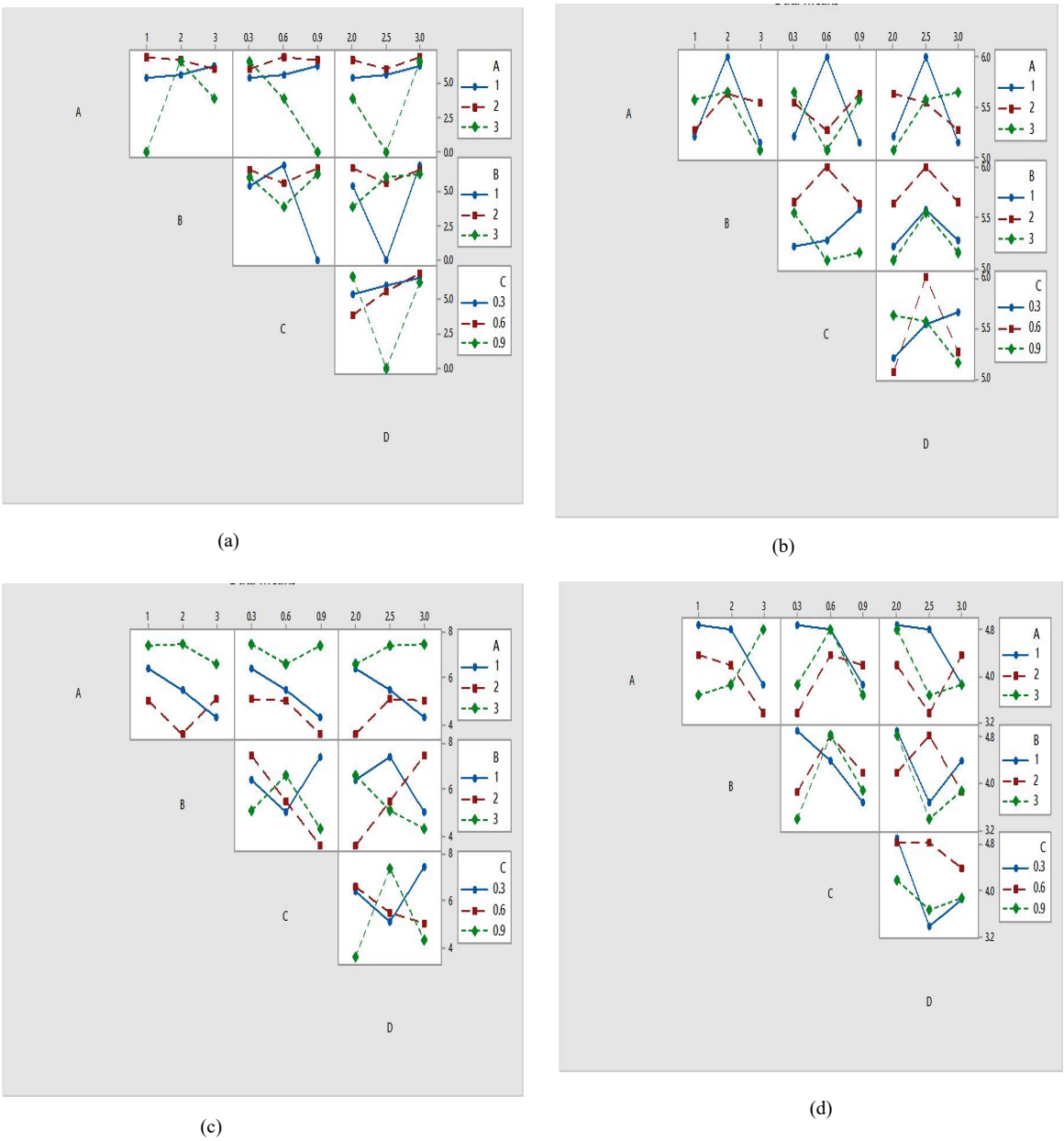
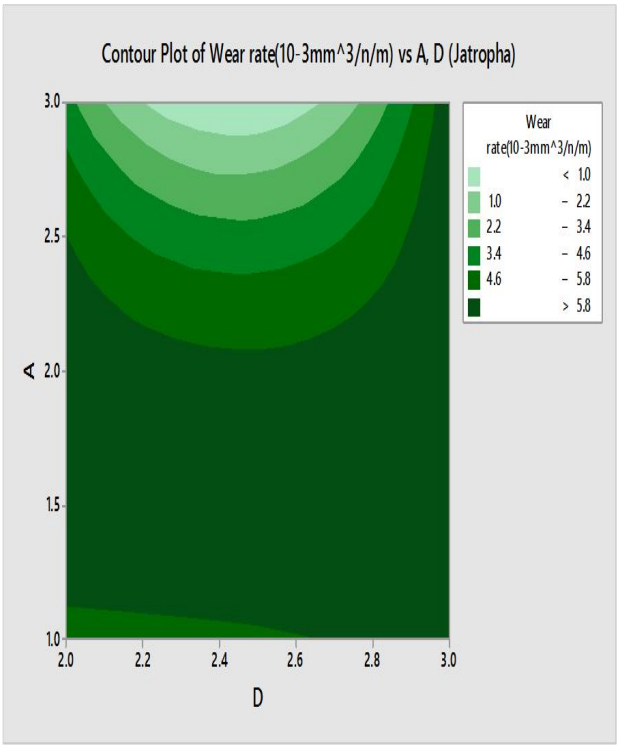
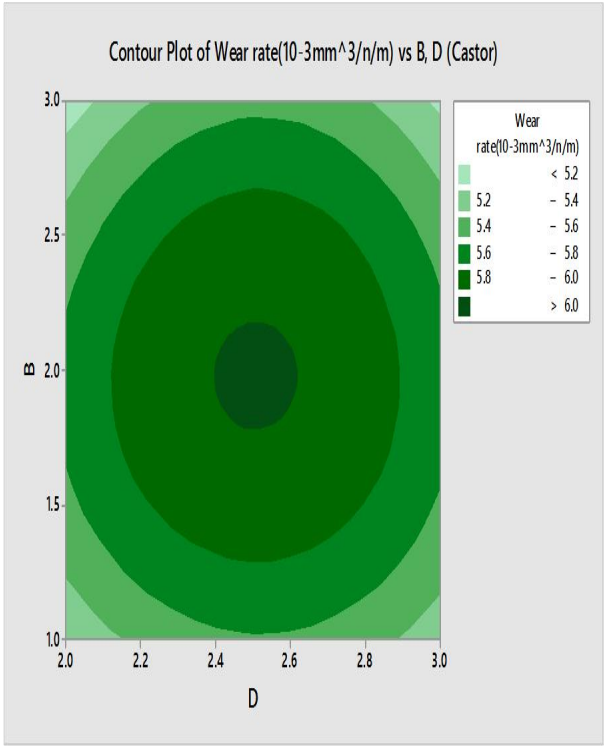


Figure 4.19: Interaction Plot of Wear Rate (a) Jatropha oil (b) Castor oil (c) Castor oil based biolubricant (d) Jatropha oil based biolubricant

The contour plots for wear rate are displayed in Figure 4.20. For the jatropha oil, when the concentration of viscosity modifier and extreme pressure additive is kept constant the following is observed: as the percentage concentration of A and D increased, the wear rate reduced. This is a desirable situation indicating that the introduction of the wear-reducing and corrosion-inhibiting additives A and D will reduce material energy loss due to wear. For the castor oil, if the concentration of the wear and high pressure controlling additives are kept constant, the following is observed: to minimise wear, the percentage weight concentration of B should be kept below 2.0% while that of D should be below 2.5%. Thus, 2% of B and 2.5% of D in castor oil will cause the highest wear rate and damage the machine parts faster. For the castor biolubricant, as the percentage concentration of A and C increases, the wear rate reduces. For the Jatropha biolubricant, lower concentration of B increased the wear rate while higher concentration of C increased wear rate. To minimise wear rate C should be limited to 0.3% while B should be increased.



(a)



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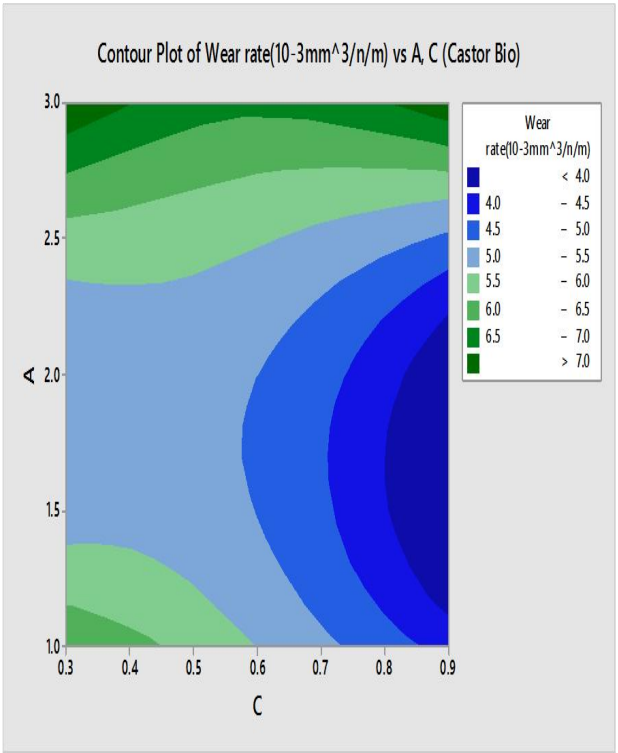

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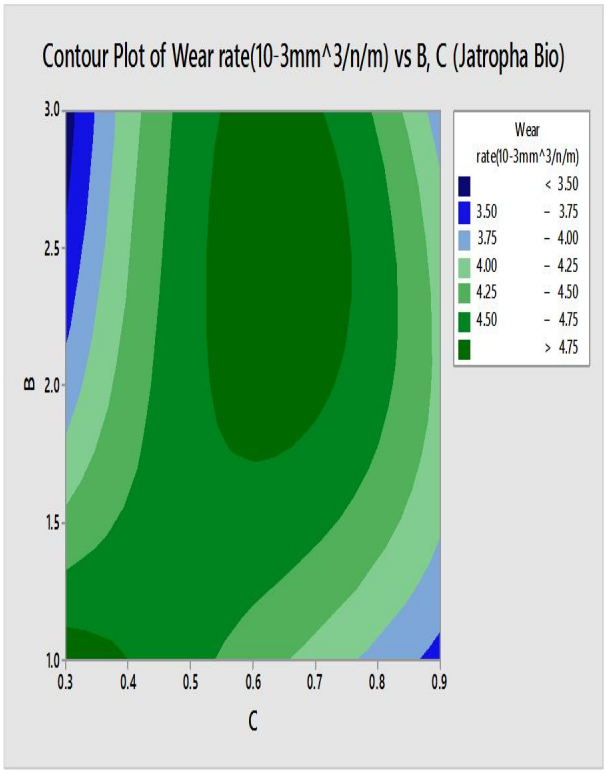


(d)

Figure 4.20: Contour Plots of Wear Rate (a) Jatropha oil (b) Castor oil (c) Castor oil based biolubricant (d) Jatropha oil based biolubricant

4.5.3 Effect of additives on the viscosity index of oils and biolubricants developed

The normal probability plot displaying the significant additives affecting the Viscosity Index (VI) for each oil and lubricant is shown in Figure 4.21. The anti-wear additive (A) had the dominant effect on the VI of the jatropha oil. The viscosity modifier additive (B) had the most significant effect on VI of the castor oil. Similarly, the anti-wear additive (A) had the most significant effect on the VI of the castor and jatropha biolubricants. It can be deduced that the anti-wear additive (A) has dominant effect on the viscosity index of jatropha and castor oil based lubricants.

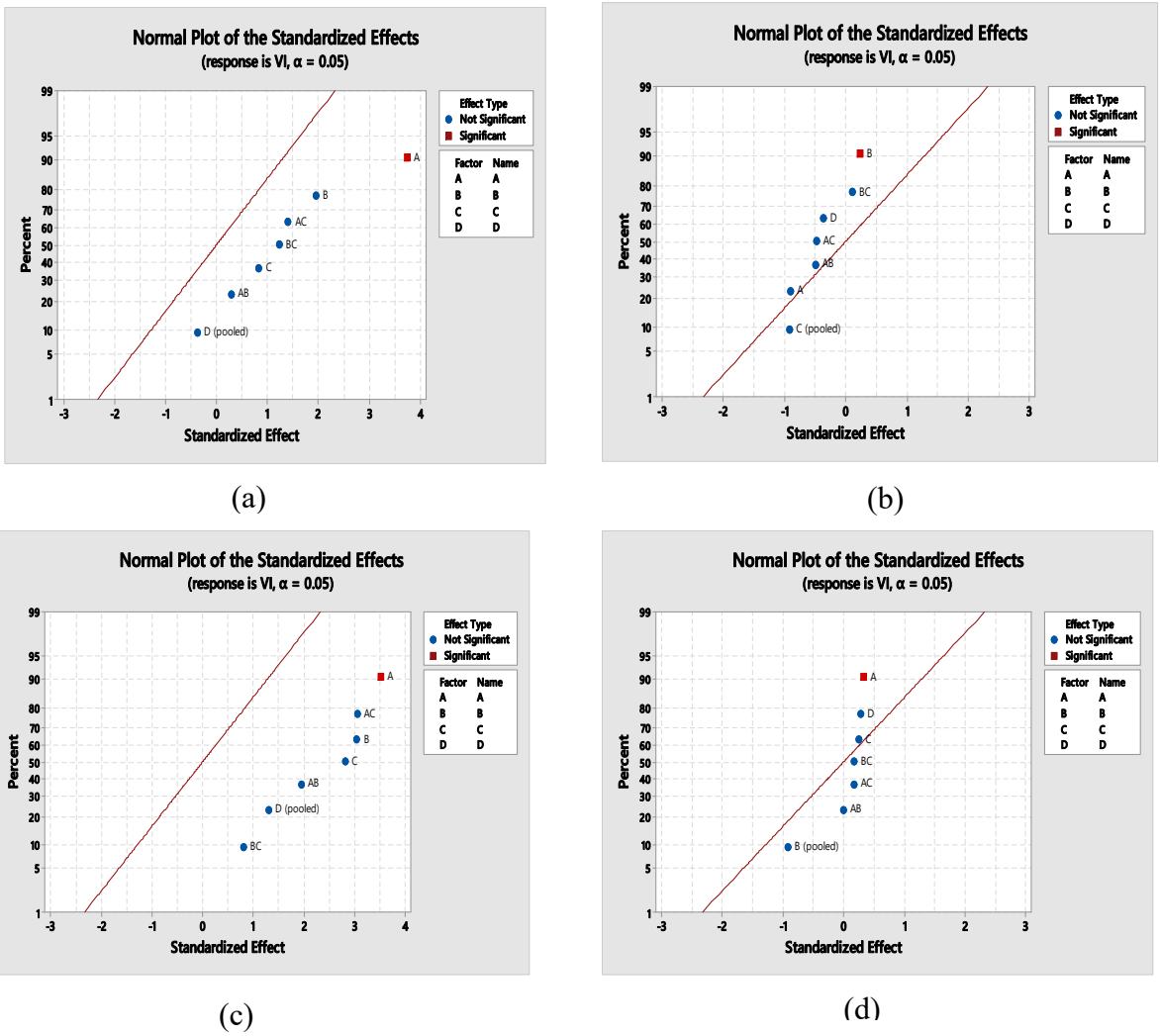


Figure 4.21: Normal Probability Plot of Viscosity Index (a) Jatropha oil (b) Castor oil (c) Castor oil based biolubricant (d) Jatropha oil based biolubricant

4.5.3.1 Analysis of variance (ANOVA) of viscosity index of jatropha and castor Oil-based lubricants

(a) Jatropha oil

The ANOVA of the VI for the jatropha oil is shown in Table 4.11a. while the most significant additive was the anti-wear additive A (80.43%) followed by the viscosity modifier additive B (11.47%). The least significant factor was the extreme pressure additive C (6.89%) while the anti-corrosion additive D contributed to the error. The error contributed 1.21% to the viscosity index.

Table 4.11a: ANOVA for Viscosity Index for Jatropha oil

Factor	DOF	SS	MS	F	P
A	2	678.8	339.4	66.2891	80.43
B	2	96.83	48.415	9.456	11.47
C	2	58.13	29.065	5.677	6.89
D	Pooled	Pooled	Pooled	pooled	pooled
Pooled Error	2	10.2400000	5.12		1.21
Total	8	844.0	105.5		100.00

(b) Castor oil

The ANOVA of the VI for castor oil is shown in Table 4.11b. The viscosity modifier B (49.9 %) had the most significant effect on the VI followed by the anti-corrosion additive D (25.91 %), and then the anti-wear additive A (23.68%). The percentage contribution of error to the viscosity index was 0.51%.

.**Table 4.11b: ANOVA for Viscosity Index for Castor oil**

Factor	DOF	SS	MS	F	P
A	2	6.55	3.275	46.1268	23.68
B	2	13.80	6.9	97.183	49.90
C	Pooled	Pooled	Pooled	pooled	pooled
D	2	7.165000	3.5825	50.458	25.91
Pooled Error	2	0.1420000	0.071		0.51
Total	8	27.7	3.457125		100.00

(c) Castor oil based biolubricant

The ANOVA of the VI for the castor oil based biolubricant is shown in Table 4.11c. The anti-wear additive A with 62.84 % contribution to VI was the most significant factor followed by the viscosity modifier additive B (20.23 %). The least significant factor was the extreme pressure additive C (15.53 %) while anti-corrosion additive D contributed to the error. The error contributed 1.39 % to the viscosity index.

Table 4.11c: ANOVA for Viscosity Index of Castor oil based biolubricant

Factor	DOF	SS	MS	F	P
A	2	1096	548	45.1029	62.84
B	2	352.90	176.45	14.523	20.23
C	2	270.9	135.45	11.148	15.53
D	pooled	Pooled	pooled	pooled	pooled
Pooled Error	2	24.3000000	12.15		1.39
Total	8	1744.1	218.0125		100.00

(d) Jatropha oil based biolubricant

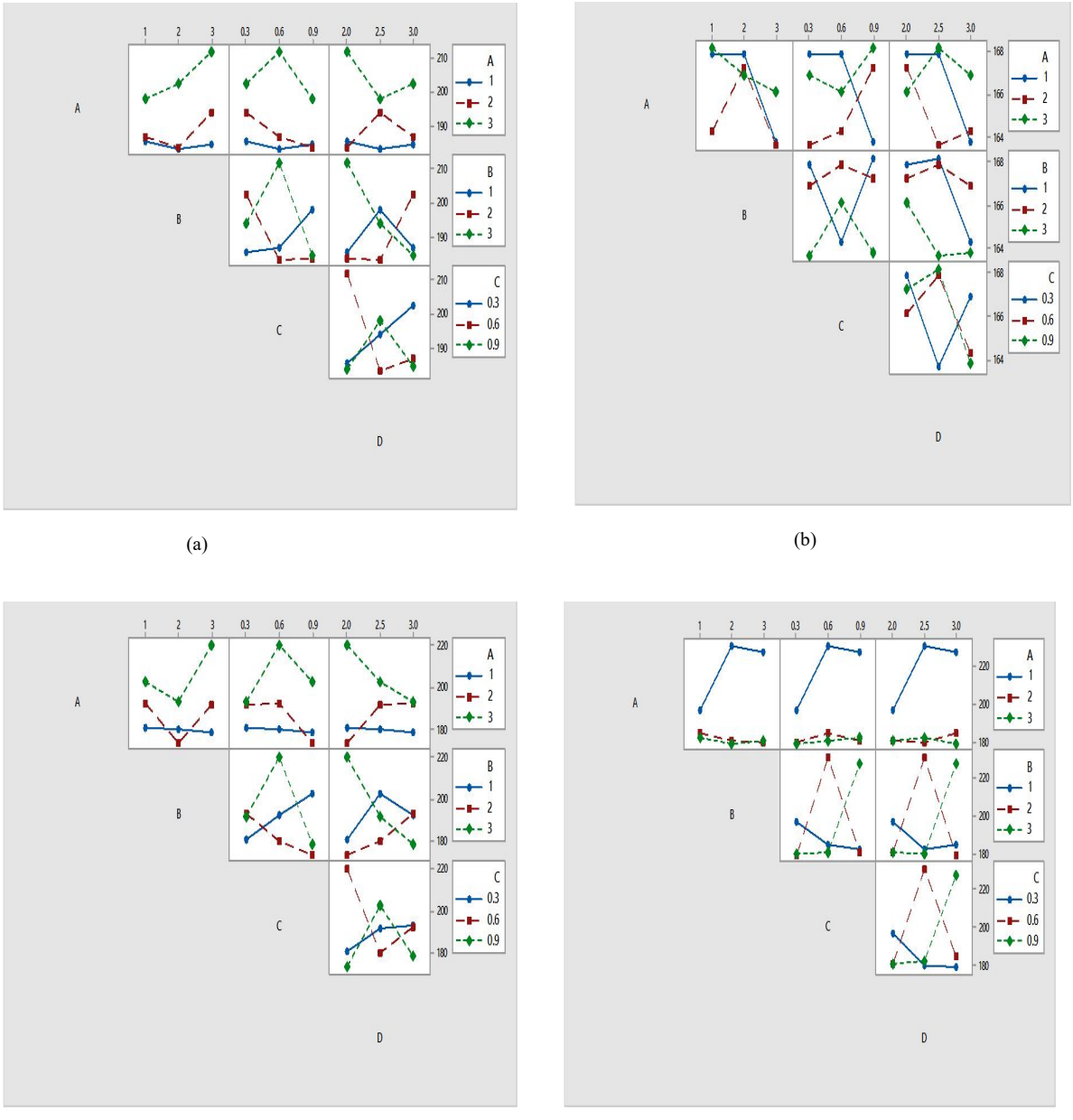
The ANOVA of the VI for the jatropha oil based biolubricant is shown in Table 4.11d. The most significant factor was the anti-wear additive A (79.33%), followed by the extreme pressure additive C (9.24%). The anti-corrosion additive D (7.37%) was the least significant factor while the viscosity modifier B contributed to the error. the error contributed 4.06% to the viscosity index.

Table 4.11d: ANOVA for Viscosity Index of Jatropha oil based biolubricant

Factor	DOF	SS	MS	F	P
A	2	2830.8	1415.4	19.5497	79.33
B	pooled	Pooled	pooled	Pooled	pooled
C	2	329.9	164.95	2.278	9.24
D	2	263.000000	131.5	1.816	7.37
Pooled Error	2	144.8000000	72.4		4.06
Total	8	3568.5	446.0625		100.00

4.5.3.2 Interactions effects on the viscosity index of oils and biolubricants developed

The interaction plot for the four parameters (percentage additives concentration) in Viscosity Index is shown in Figure 4.22. The plots shows that there was an interaction between the additives A, B, C and D at all levels. This is in agreement with the findings of earlier researchers that additives interact with each other and some of the interactions can be either destructive or enhancing (Ratoi *et al*., 2000; Menezes *et al.,* 2013).



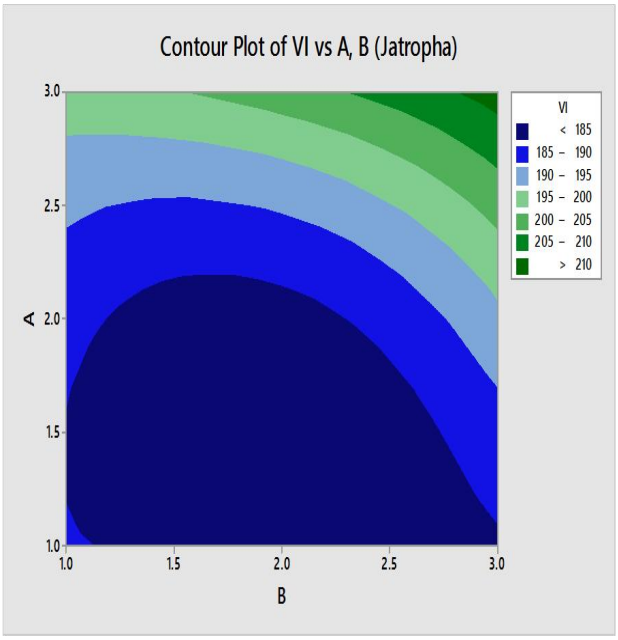
(a) (b)

(c) (d)

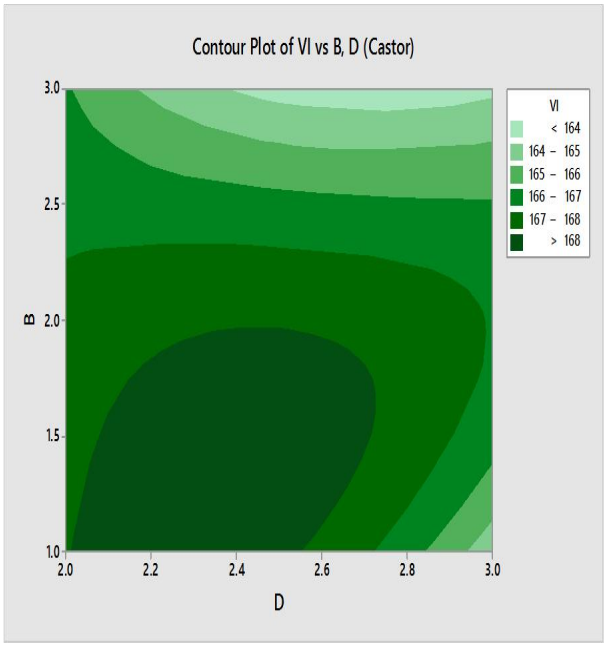
Figure 4.22: Interaction Plot of Viscosity Index (a) Jatropha oil (b) Castor oil (c) Castor oil based biolubricant (d) Jatropha oil based biolubricant

The contour plots for viscosity index of the jatropha and castor oil base lubricant are displayed in Figure 4.23. For the jatropha seed oil, as the percentage concentration of A increases, the VI also increased while the VI remained constant for all concentrations of B. Thus, the VI depends majorly on the concentration of A. It can be concluded that the viscosity modifier additive (HEC) concentration has little effect on the VI of jatropha oil. Conversely, higher concentration of the antiwear additive (TCP) improves the VI of jatropha oil. For the castor oil, as the concentration of B and D both increases, the VI decreased. It should be noted that castor oil has low VI and the use of additives especially HEC (above 2%) and ZnDDC (above 2.7%) further damages the VI of castor oil.

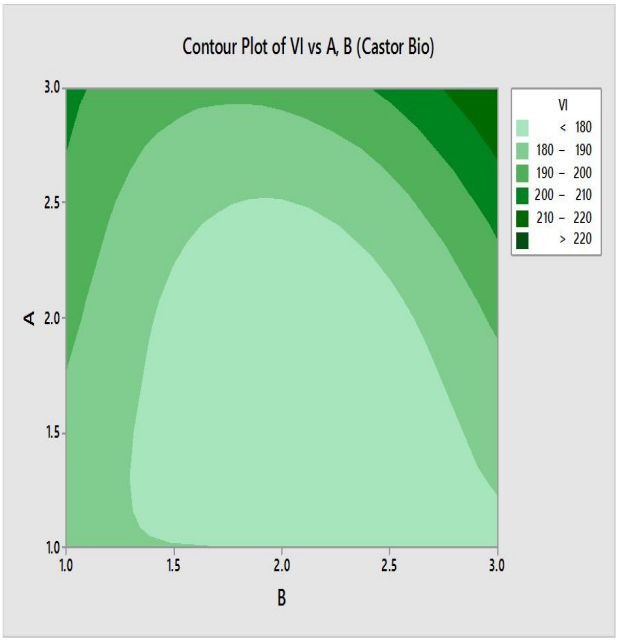
For, the castor biolubricant, as the concentration of the viscosity modifier B increases above 1%, the lower the VI. This is similar to the findings of Quinchia *et al.* (2013) and also, as the concentration of the anti-wear additive A increases so also the VI of castor biolubricant increased. For the jatropha biolubricant, the higher the quantity of anti-wear additive A, the lower the VI and the higher the concentration of the extreme pressure additive C, the higher the VI. For good VI in jatropha biolubricant the concentration of anti-wear A should not exceed 1% while the extreme pressure additive C should not be below 0.6 %.



(a)



(b)



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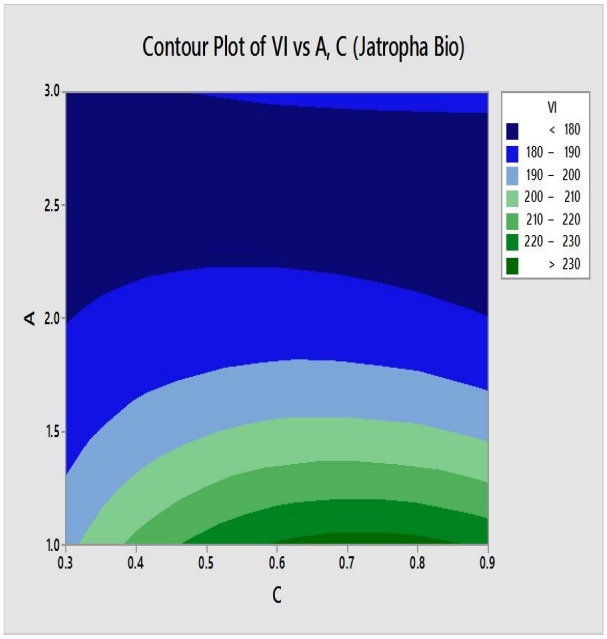

(d)

Figure 4.23: Contour Plots of Viscosity Index (a) Jatropha oil (b) Castor oil (c) Castor biolubricant (d) Jatropha biolubricant

CHAPTER FIVE

5.0 CONCLUSION AND RECOMMENDATIONS

5.1 Conclusion

The tribological evaluation of lubricants developed from jatropha and castor oils for industrial applications has been successfully carried out in this study. In conclusion the developed jatropha and castor oil base lubricant are suitable replacement for the environmentally hostile mineral based lubricants for industrial applications. The detail conclusions drawn from this research are stated in subsections 5.1.1-5.1.4.

5.1.1 Characterisation and modification of jatropha and castor oil

The castor oil is denser than the jatropha oil while both oils are denser than the SAE 20W/50 but less dense compared to water and would therefore float in water. Both jatropha and castor oils have good lubricant properties but were deficient in their wear, long storage and thermo-oxidative stabilities. These limitations may be due to the high levels of unsaturation in the oils and the high level of free fatty acids in the oils. Both oils had excellent corrosion inhibition properties and were of corrosion grade 0.

Modification of the jatropha and castor oils by acid catalysed esterification improved their properties. The modified oils had better cold flow properties, improved thermal and oxidative stability. The modified oils were also of corrosion grade 0 but were of less viscosity and flash points. The modified and unmodified jatropha and castor oils are highly biodegradable while the SAE 20W/50 had very poor biodegradability.

5.1.2 Development of jatropha and castor oil based biolubricants

The developed biolubricants had excellent physicochemical, rheological, temperature and thermo-oxidative properties. The biolubricants would have longer shelf life.

These biolubricants developed from jatropha and castor oils have performance comparable to that of mineral oil base lubricant SAE 20/W50. Both bioluricants had alkaline pH, high viscosity index, appreciable viscosity and excellent cold flow properties. The biolubricants produced are of excellent corrosion inhibition properties and are of corrosion grade 0.

The biodegradability of the developed jatropha and castor oils based biolubricants are similar to those of the modified and unmodified jatropha and castor oils. The developed biolubricants had far better biodegradability than the SAE 20W/50.

5.1.3 Tribological evaluation of jatropha and castor oils base lubricants

The jatropha oil performed better than the SAE 20W/50 in friction reduction and wear prevention. The castor oil is better than the SAE 20W/50 in friction reduction while the SAE 20W/50 is better than the castor oil in wear prevention. The jatropha oil is a better lubricant compared to the castor oil.

Modified jatropha oil has better friction reduction but lower wear prevention properties than the SAE 20/W50. The modified castor oil has better friction reduction and wear prevention properties compare to SAE 20W/50. The modification of jatropha oil through acid catalysed esterification reduces its friction and wear performance. The modification of castor oil improves its friction reduction and wear prevention properties.

The developed jatropha oil biolubricant was found to be better in frictional performance than SAE 20W50 and had similar wear performance with that of SAE 20W/50. The developed castor oil biolubricant exceeded the SAE 20W/50 both in friction and wear performance. The castor biolubricant is better than the jatropha oil biolubricant in tribological performance. The jatropha oil biolubricant has lower tribological performance compared to jatropha oil while castor biolubricant has better tribological performance than castor oil.

5.1.4 Determination of the influence of additives on the tribological behaviour of the developed jatropha and castor oil based biolubricants

The concentration of the extreme pressure additive cetyl chloride has dominant effect on the friction response of developed jatropha and castor oil bio-lubricants. Anti-wear additive Tricysl Phosphate exerts a dominating effect on the viscosity index of the jatropha and castor oil based lubricants. The viscosity modifier hydroxy ethyl cellulose has very little effect on the viscosity of the jatropha oil while higher concentration of the anti-wear additive improves the viscosity index of the jatropha oil. There was an interaction between the four additives that influence the tribological performance of the jatropha and castor oil based lubricants.

5.2 Recommendations

The research was carried out using laboratory equipment that are of small sizes, further research can be carried out in the aspect of the design of suitable reactors that can be used for mass production of the biolubricants. A robust design that will enable automated continuous production of the biolubricant will be a step in the right direction.

Additionally, the catalyst and additives used were laboratory grade chemicals which are expensive and mostly imported. Research into new local materials that can produce the same or superior results is here by recommended. Additionally, alternative chemical or thermal modification should be considered. Also, research efforts towards production of biolubricant from a blend of jatropha and castor oil will be another interesting aspect. Blending the biolubricant and mineral oil based lubricant should be looked into. Alternatively, research into the use of modified jatropha and/or castor oil as antifriction additive in mineral oil based lubricant and the resulting effect on its biodegradability is highly recommended.

5.3 Contribution to Knowledge

This research has:

(i) Demonstrated that jatropha and castor oil unlike other vegetable oils possess excellent cold flow properties which makes them better suited for the production of industrial lubricants.

(ii) Developed jatropha and castor oil based biolubricants with improved performance and properties comparable to mineral oil based lubricant SAE 20/W50.

(iii) Determined additives that have the most significant effect and non-dominant effect on the tribological output of developed jatropha and castor biolubricants.

(iv) Produced data on the tribological evaluation of lubricants developed from Nigeria produced jatropha and castor oils.

(v) Provided numerical values for the biodegradability of jatropha oil (81 %) and castor oil (96 %) derived lubricants and mineral oil based lubricant SAE 20/W50 (34 %).

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APPENDIX A

Publications

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Review

Vegetable Oil Based Lubricants: Challenges and Prospects

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Abstract

Lubricants are very important consumables in all industries as failure in machine parts due to absence or wrong choice of lubricants carries enormous cost. The base oil used for the formulation of most lubricants is environmentally hostile mineral oil and 30% of lubricants consumed ends up in the ecosystem. However, mineral oil reserve is depleting and the environmental concern about the damaging impact of mineral oil is growing. The search for environment friendly substitutes to mineral oils as base oils in lubricants has become a frontier area of research in the lubricant industry. Vegetable oils are perceived to be alternatives to mineral oils for lubricant base oils due to certain inherent technical properties and their ability to be biodegradable. This paper is an overview of recent research on vegetable oils as base oil for lubricant production with focus on the prospects, challenges and efforts to overcome the challenges of using vegetable oils as base oil for the production of industrial lubricants. Compared to mineral oils, vegetable oils in general possess high flash point, high viscosity index, high lubricity, low evaporative loss, are renewable, and are environmentally friendly. Poor oxidative and hydrolytic stability, high cost, food versus energy debate, high temperature sensitivity of tribological behaviour and poor cold flow properties are reckoned to be the limitations of vegetable oils for their use as base oils for industrial lubricants. The current effort to overcome these limitations includes the use of non edible oils, additives, chemical modifications and thermal modifications. More research and legislation in favour of the use of vegetable oil lubricants is recommended.

Keywords

vegetable oil, biolubricants, environmentally-friendly, wear, friction, tribometers

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Nigeria Jatropha oil as suitable basestock for biolubricant production

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KEYWORDS

Jatropha oil
Biolubricant
Viscosity Index
Tribology
Cold flow
Environmentally-friendly

ABSTRACT

Jatropha oil which is a non-edible vegetable oil that is sustainable, biodegradable and environmentally friendly is thought to be a good substitute for mineral oil for lubricant production. The physicochemical, rheological, temperature, thermo-oxidative stability and corrosion properties of Nigerian Jatropha oil and commercially available mineral oil base lubricant (SAE 20W50) were determined for suitability as base stock for lubricant production. The Jatropha oil has specific gravity of 0.91, free fatty acid of 15.6 mg KOH/g , pH of 5.82, saponification value of 220.46 mg KOH/g and Iodine value of 88.9 gI2/100g oil. Assessment of the rheological and temperature properties of the Jatropha oil gave kinematic viscosity at 400C and 1000C as 83.2 cSt and 63.5 cSt respectively, viscosity index of 145.5, pour point of -11.20C, cloud point of -8.30C and flash point of 2640C. The peroxide value of the Jatropha oil was 5.98 meq/Kg and it was of corrosion grade 0. The jatropha oil has better viscosity index compared to the SAE 20W50, whereas the SAE 20W50 is better than the jatropha oil in other measured properties. The properties of the Jatropha oil need to be improved except its cold flow, flash point and corrosion inhibition properties.

The Nigerian Society of Engineers
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Conference theme
Role of Engineering in Sustainable Development Goals

OXIDATIVE STABILITY AND COLD FLOW PROPERTIES OF NON-EDIBLE VEGETABLE OIL FOR INDUSTRIAL BIOLUBRICANT APPLICATIONS

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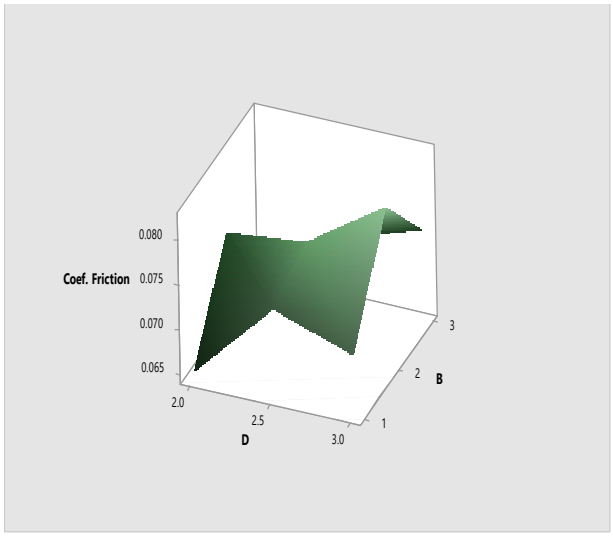
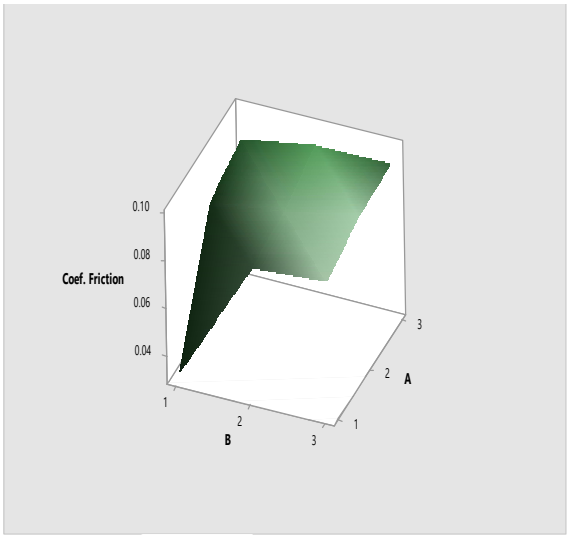
ABSTRACT

The environmental concern about the use of mineral oil-based lubricants is increasing. Additionally, mineral oil is non-renewable and subject to high price fluctuation. There is therefore a need to seek alternative sustainable and renewable lubricant base stock. Vegetable oil are thought of as good substitute for mineral oil -based lubricants, however their use is limited by their poor cold flow properties and poor thermo-oxidative stability. The oxidative stability, flash point and

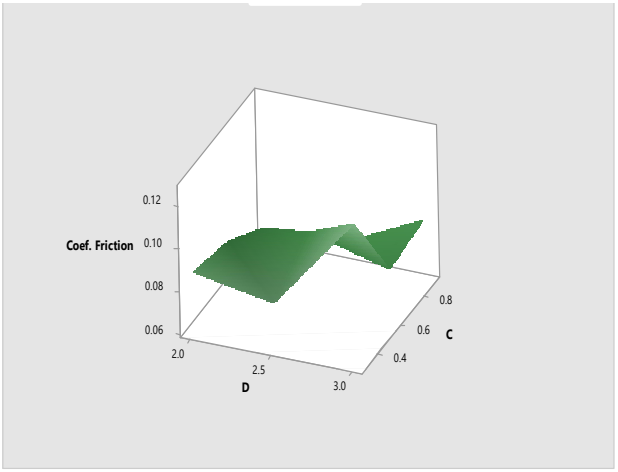
APPENDIX B
3-D Surface Plots



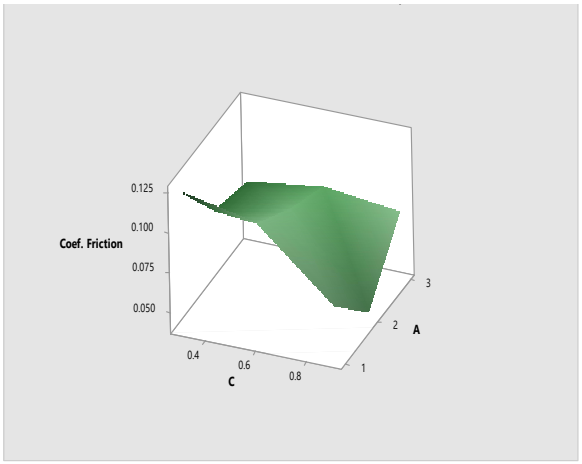
(a)



(b)

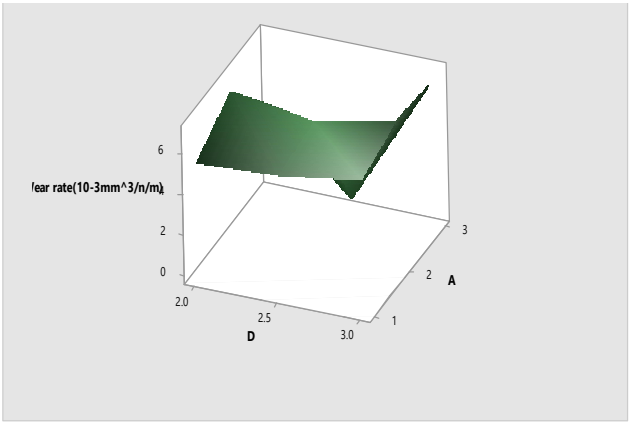


(c)

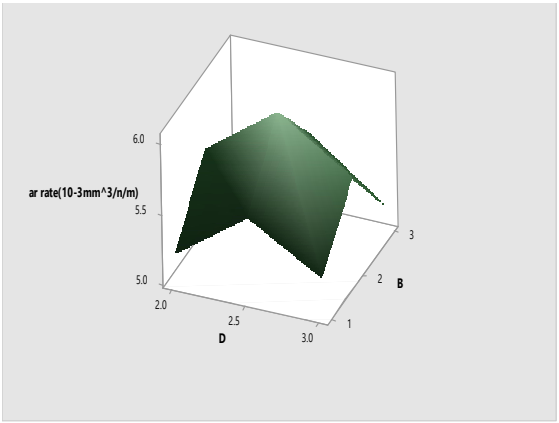


(d)

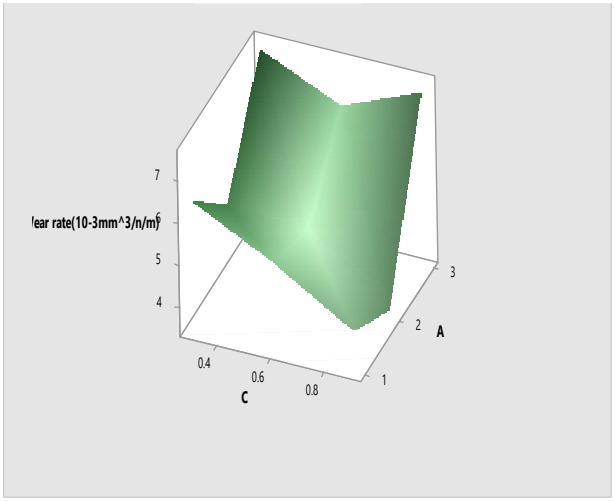
Figure B1: 3-D surface plots oils and developed biolubricants for coefficient of friction (a) jatropha oil (b) castor oil (c) castor oil based biolubricant (d) jatropha oil based biolubricant



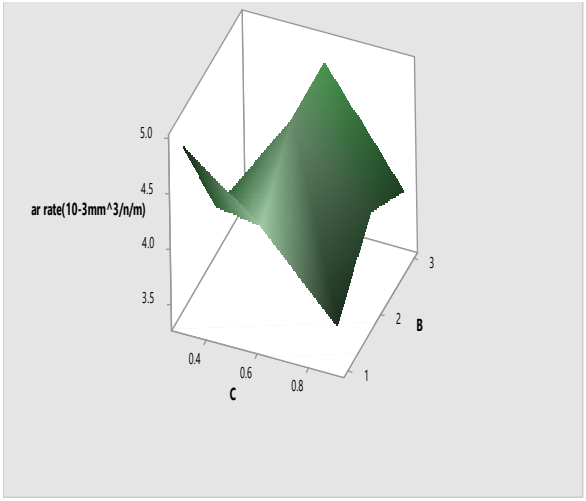
(a)



(b)

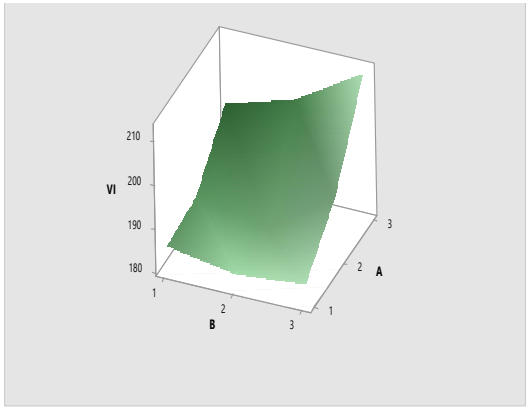


(c)

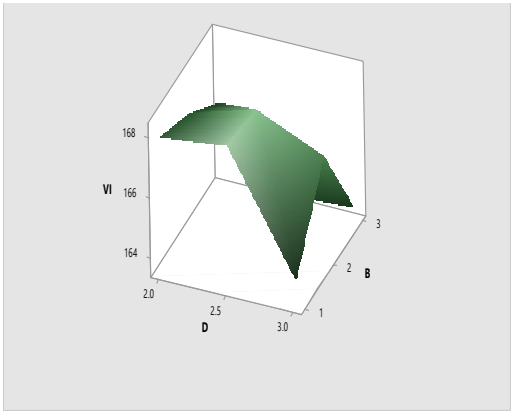


(d)

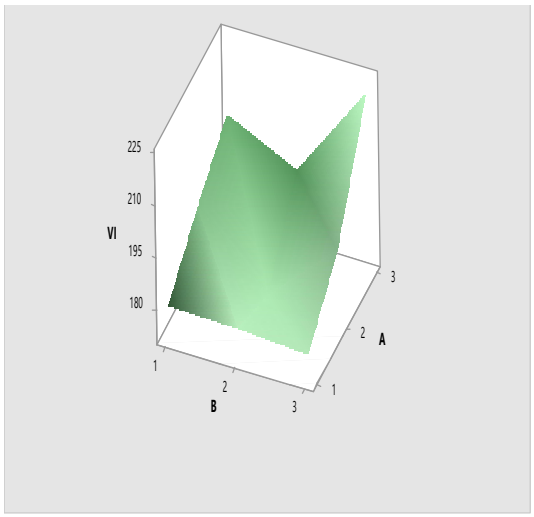
Figure B2: 3-D surface plots oils and developed biolubricants for wear rate (a) jatropha oil (b) castor oil (c) castor oil based biolubricant (d) jatropha oil based biolubricant



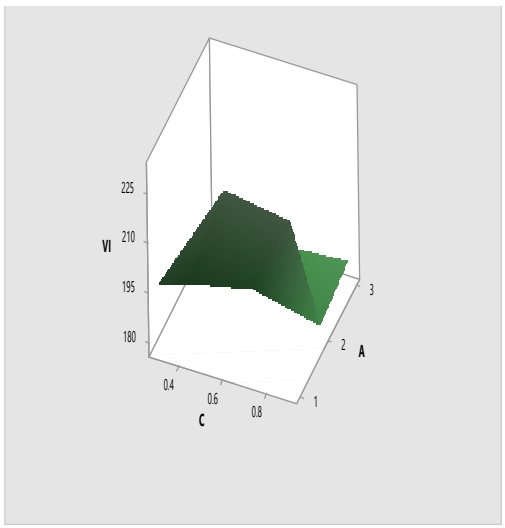
(a)



(b)



(c)



(d)

Figure B3: 3-D surface plots oils and developed biolubricants for viscosity index (a) jatropha oil (b) castor oil (c) castor oil based biolubricant (d) jatropha oil based biolubricant

APPENDIX C
Tribological Test Result

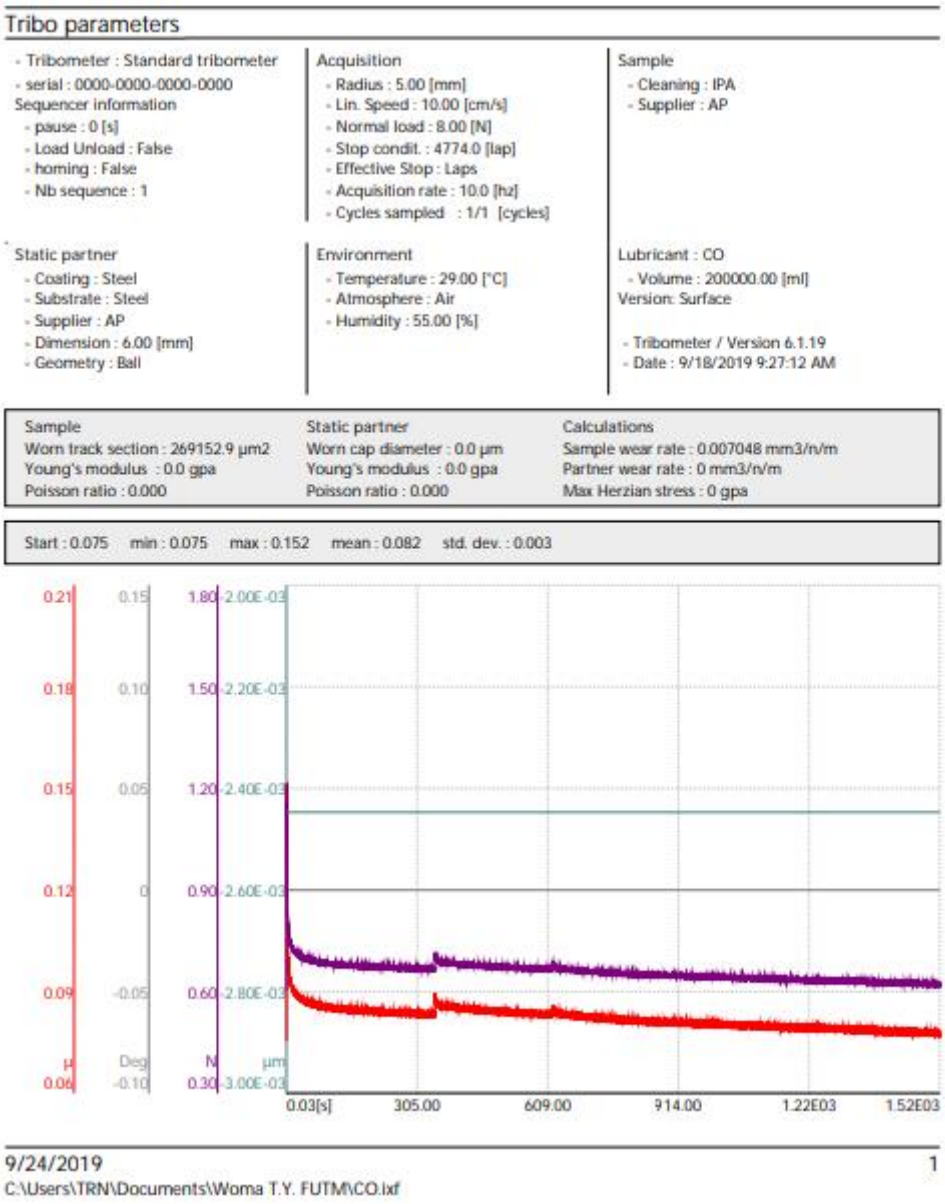


Figure C1: Tribological test result for castor oil

Tribo measurement1

Tribo parameters

- Tribometer : Standard tribometer
- serial : 0000-0000-0000-0000

Sequencer information
- pause : 0 [s]
- Load Unload : False
- homing : False
- Nb sequence : 1

Acquisition
- Radius : 5.00 [mm]
- Lin. Speed : 10.00 [cm/s]
- Normal load : 8.00 [N]
- Stop condit. : 4774.0 [lap]
- Effective Stop : Laps
- Acquisition rate : 10.0 [hz]
- Cycles sampled : 1/1 [cycles]

Sample
- Cleaning : IPA
- Supplier : AP

Static partner
- Coating : Steel
- Substrate : Steel
- Supplier : AP
- Dimension : 6.00 [mm]
- Geometry : Ball

Environment
- Temperature : 29.00 [°C]
- Atmosphere : Air
- Humidity : 55.00 [%]

Lubricant : ECO
- Volume : 200000.00 [ml]

Version: Surface
- Tribometer / Version 6.1.19
- Date : 9/18/2019 11:06:06 AM

Sample	Static partner	Calculations
Worn track section : 236763.1 µm2	Worn cap diameter : 0.0 µm	Sample wear rate : 0.0062 mm3/n/m
Young's modulus : 0.0 gpa	Young's modulus : 0.0 gpa	Partner wear rate : 0 mm3/n/m
Poisson ratio : 0.000	Poisson ratio : 0.000	Max Herzian stress : 0 gpa

Comments

Start : -0.007	min : -0.007	max : 0.176	mean : 0.078	std. dev. : 0.009

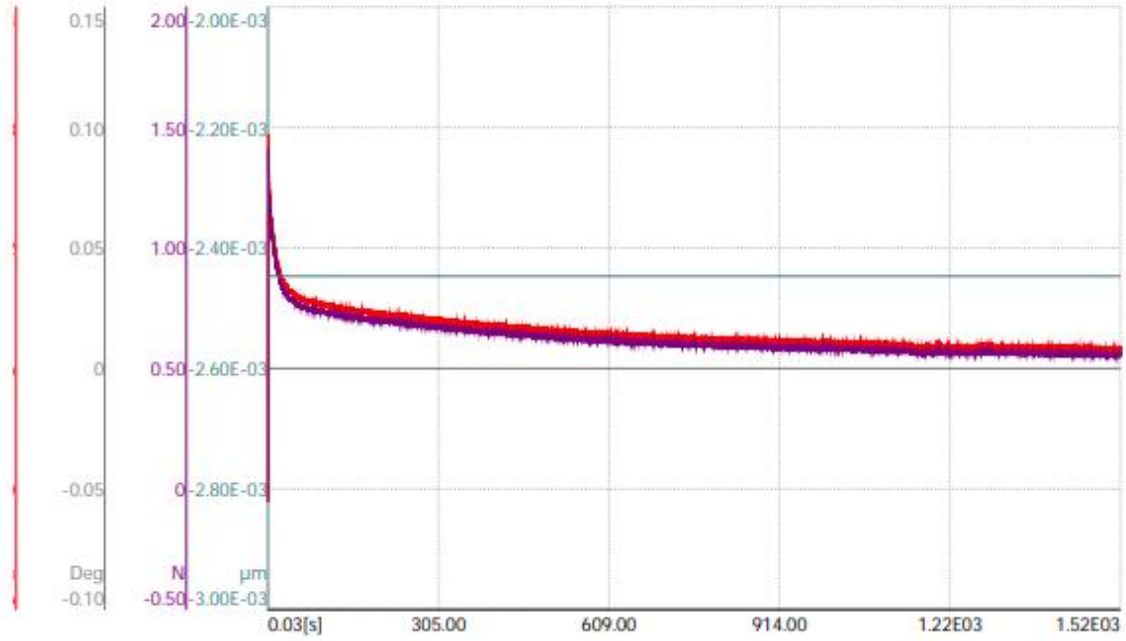


Figure C2: tribological test result for modified castor oil

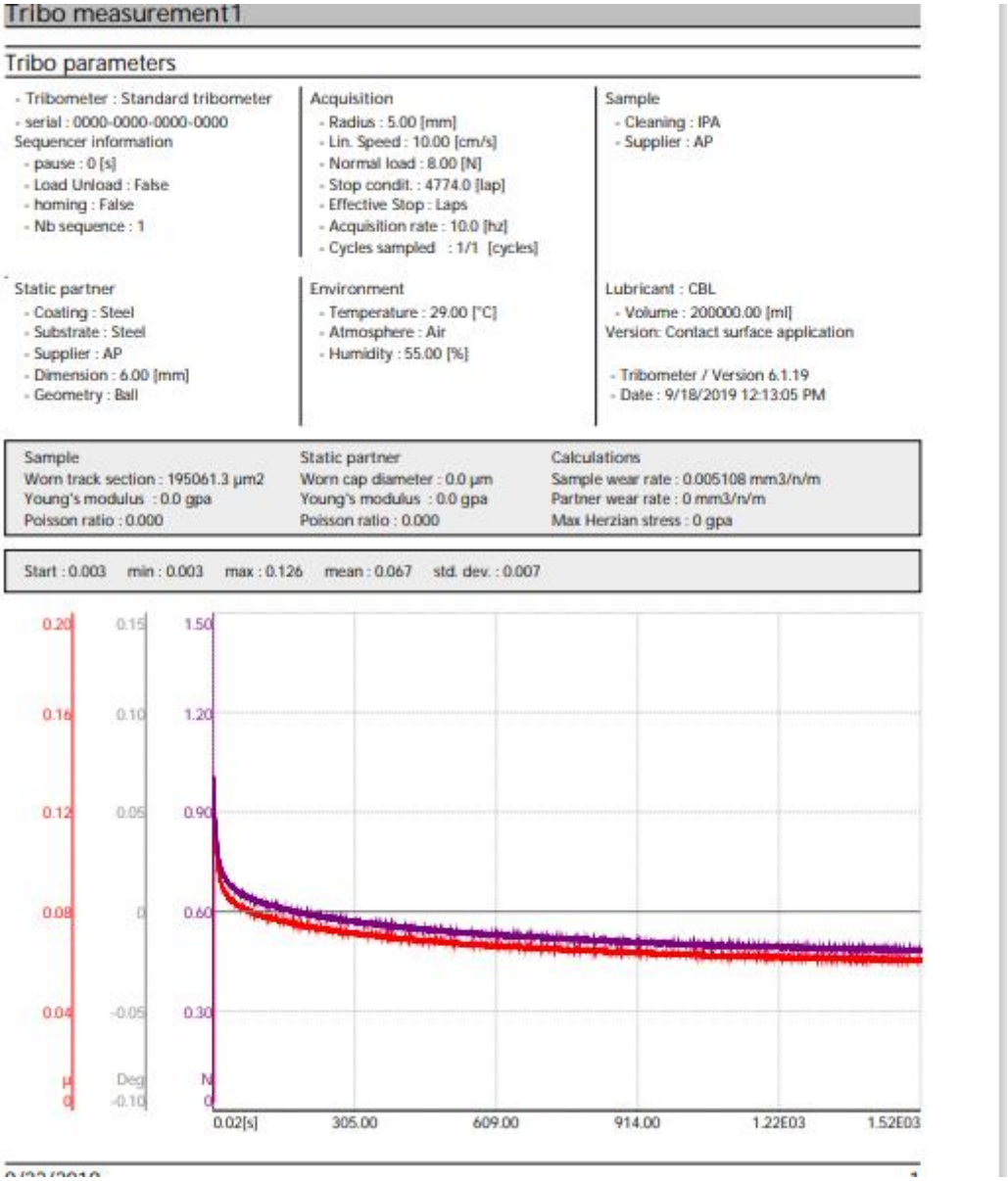


Figure C3: tribological test result for castor biolubricant

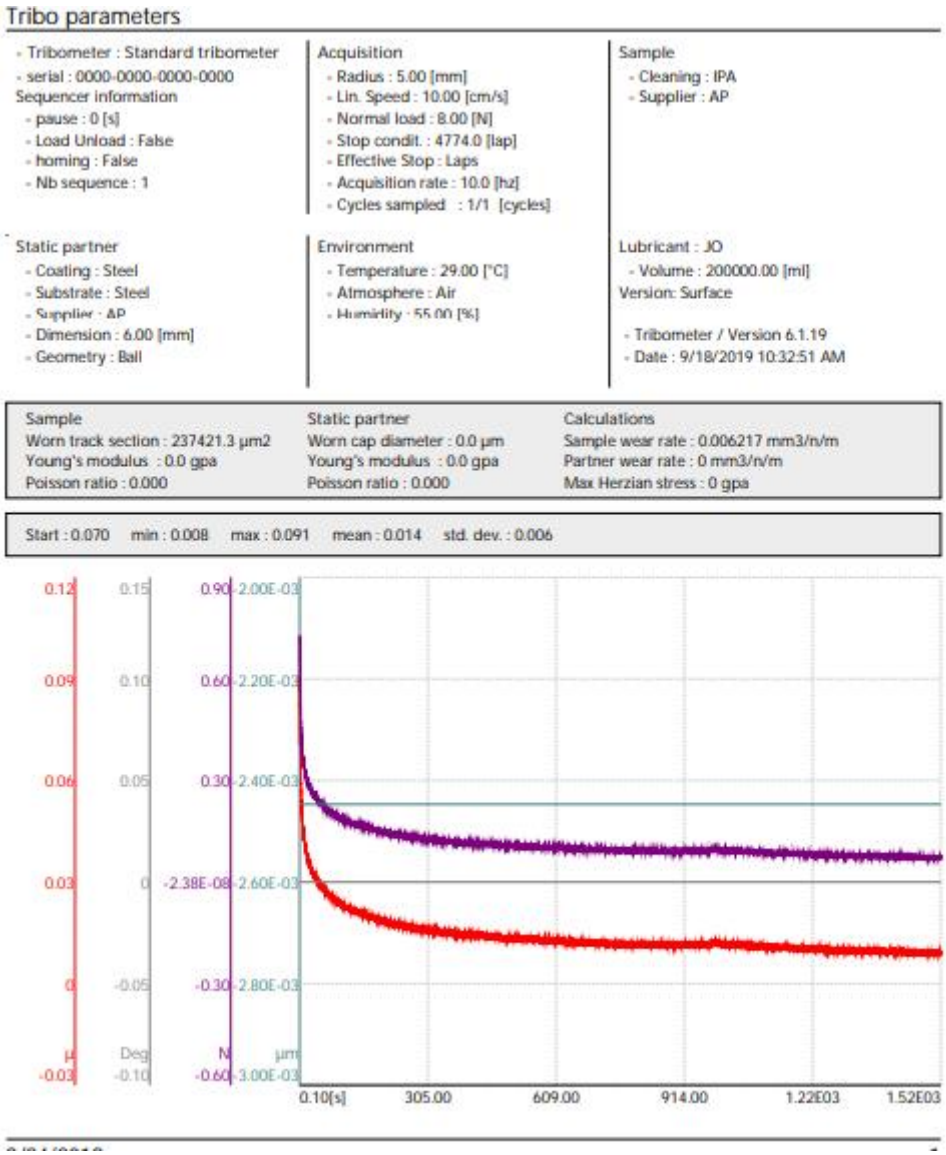


Figure C4: Tribological test result for jatropha oil

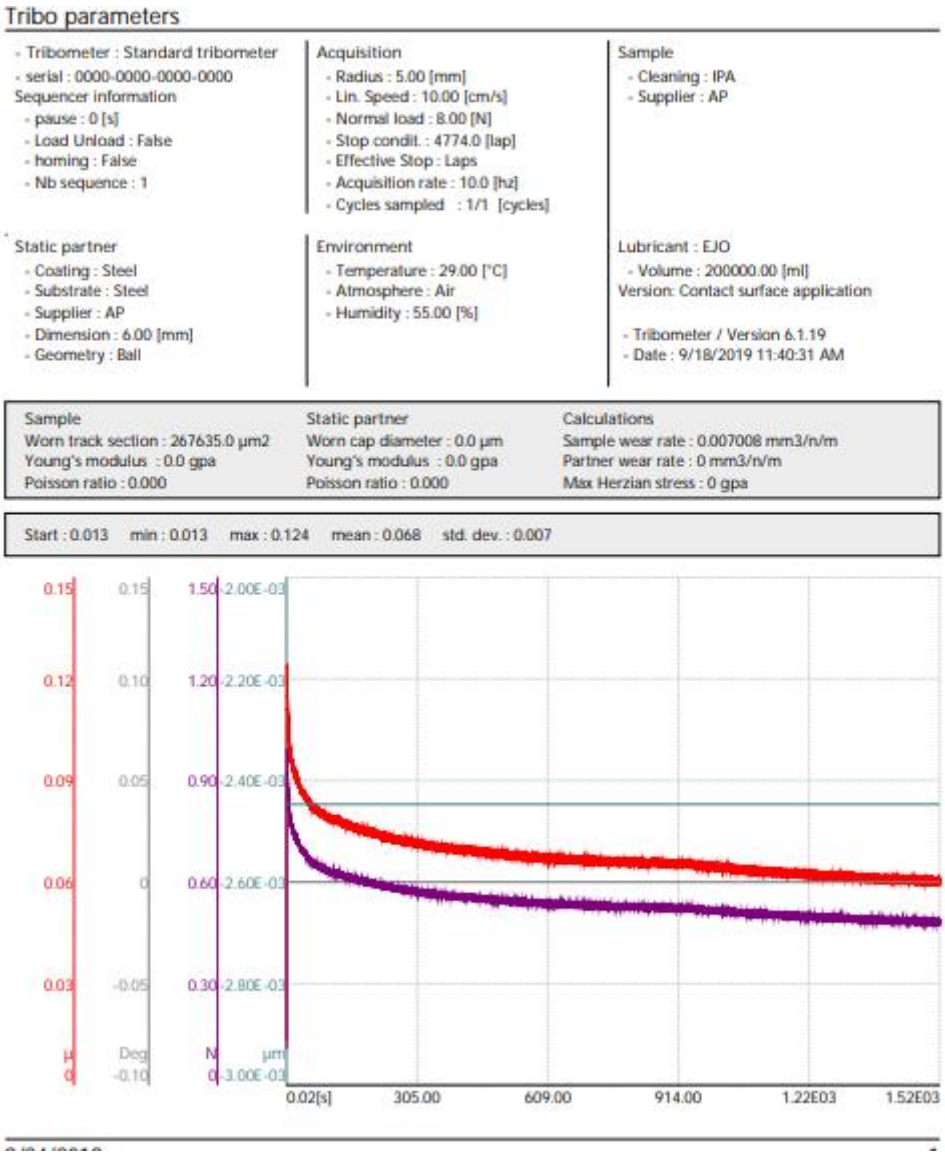


Figure C5: Tribological test result for modified jatropha oil

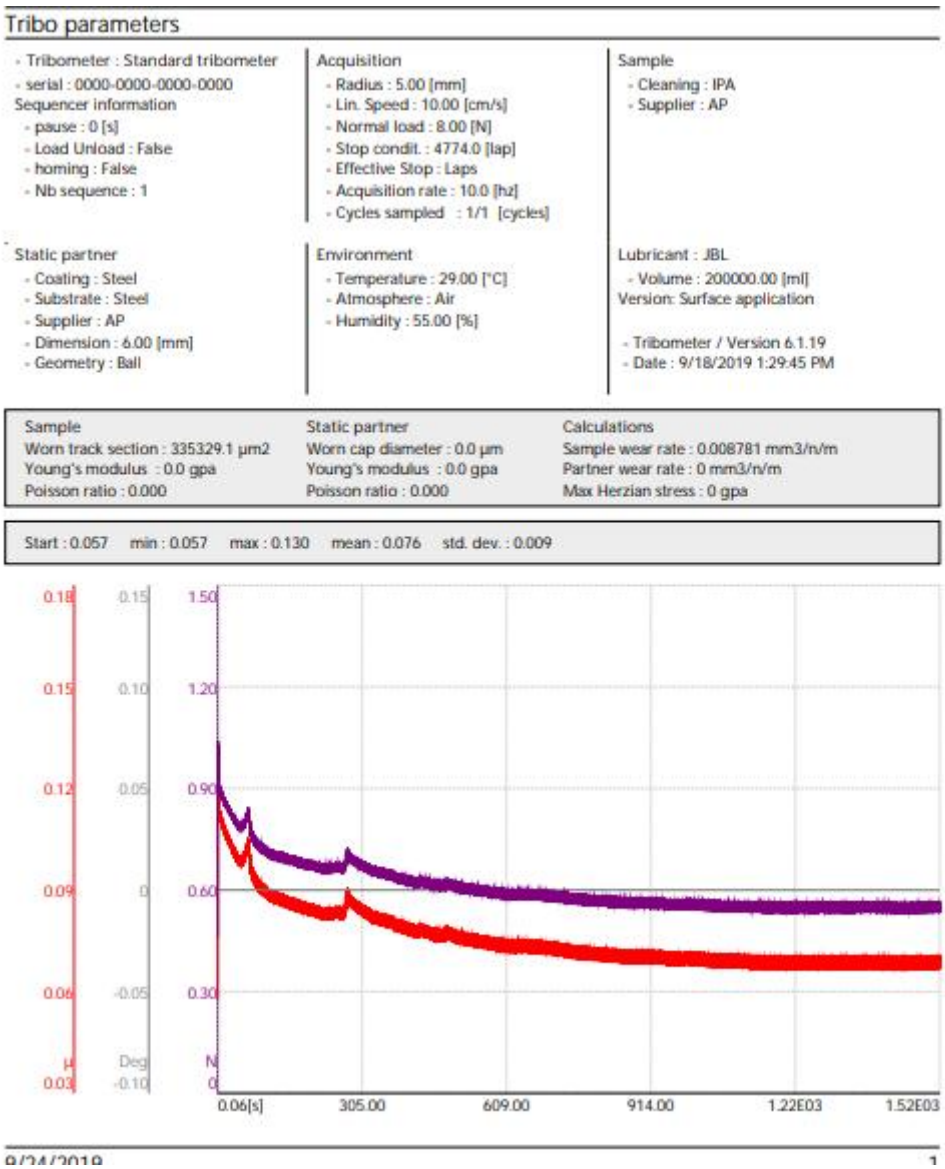


Figure C6: Tribological test result for jatropha biolubricant

Tribo measurement1

Tribo parameters

- Tribometer : Standard tribometer
- serial : 0000-0000-0000-0000

Sequencer information
- pause : 0 [s]
- Load Unload : False
- homing : False
- Nb sequence : 1

Acquisition
- Radius : 5.00 [mm]
- Lin. Speed : 10.00 [cm/s]
- Normal load : 8.00 [N]
- Stop condit. : 4774.0 [lap]
- Effective Stop : Laps
- Acquisition rate : 10.0 [hz]
- Cycles sampled : 1/1 [cycles]

Sample
- Cleaning : IPA
- Supplier : AP

Static partner
- Coating : Steel
- Substrate : Steel
- Supplier : AP
- Dimension : 6.00 [mm]
- Geometry : Ball

Environment
- Temperature : 29.00 [°C]
- Atmosphere : Air
- Humidity : 55.00 [%]

Lubricant : MNL
- Volume : 200000.00 [ml]

Version: Surface

- Tribometer / Version 6.1.19
- Date : 9/18/2019 12:42:56 PM

Sample	Static partner	Calculations
Worn track section : 255960.9 µm2	Worn cap diameter : 0.0 µm	Sample wear rate : 0.006702 mm3/n/m
Young's modulus : 0.0 gpa	Young's modulus : 0.0 gpa	Partner wear rate : 0 mm3/n/m
Poisson ratio : 0.000	Poisson ratio : 0.000	Max Herzian stress : 0 gpa

Comments

Note: both the Friction coefficient and the friction force merged. Hence the reason why only the Fricton coefficient line is shown.

Start : -0.004 min : -0.004 max : 0.139 mean : 0.115 std. dev. : 0.002

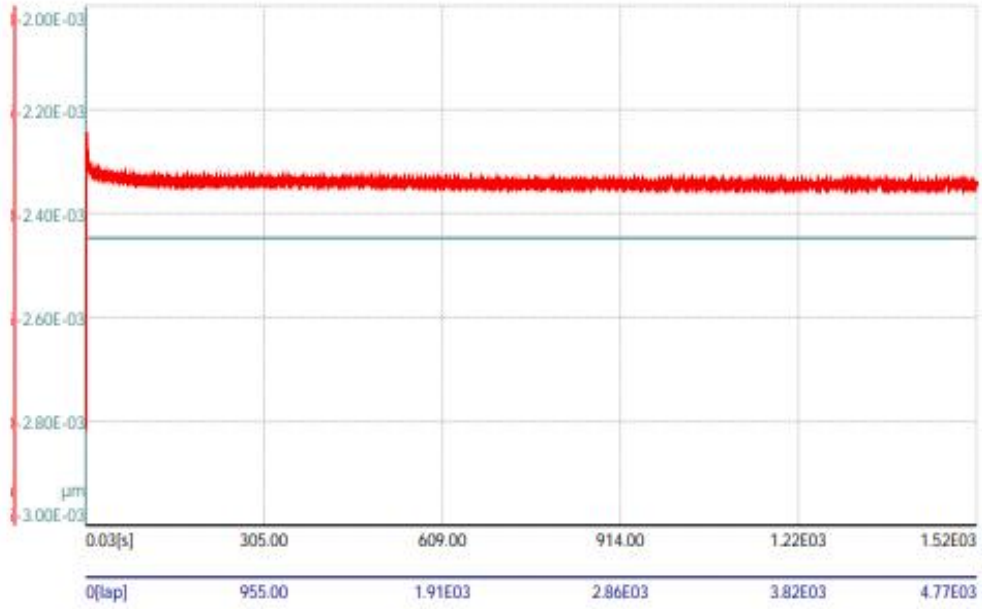


Figure C7: Tribological test result for mineral oil lubricant SAE 20/W50

APPENDIX D

Evaluation of Proportion of Additive Package for Lubricant Formulation

For run 1:

Total percentage of additive package in lubricant = 1+1+0.3+2 = 4.3 wt%

Mass of additive package in 50 g lubricant = (4.3/100) X 50 g = 2.15 g

Mass of additive A in additive package =0.5 g

A% in additive package = (0.5/2.15) X 100 = 23.25 wt%

Mass of additive B in additive package =0.5 g

B% in additive package = (0.5/2.15) X 100 = 23.25 wt%

Mass of additive C in additive package =0.15 g

C% in additive package = (0.15/2.15) X 100 = 7.00 wt%

Mass of additive D in additive package = 1.00 g

D% in additive package = (1.0/2.15) X 100 = 46.5 wt%

Table D1: Proportion of Additive Package Used

RUN	Additive package		Proportion of Additives in additive package			
	Proportion in lubricant (%)	Mass (%)	A (%)	B (%)	C (%)	D (%)
1	4.30	2.15	23.25	23.25	7.00	46.50
2	6.10	3.05	16.40	32.80	9.80	41.00
3	7.90	3.95	12.60	38.00	11.40	38.00
4	6.60	3.30	30.30	15.20	9.10	45.40
5	6.90	3.45	29.00	29.00	13.00	29.00
6	7.80	3.90	25.60	38.50	3.80	32.10
7	7.40	3.70	40.50	13.50	12.20	33.80
8	8.30	4.15	36.10	24.20	3.60	36.10
9	8.60	4.30	34.90	34.90	7.00	23.20