CERTIFICATION OF APPROVAL

Viscosity Studies Of Synthesized Polyol Ester From Jatropha Oil

by

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CERTIFICATION OF ORIGINALITY

This is to certify that I am responsible for the work submitted in this project, that the original work is my own except as specified in the references and acknowledgements, and that the original work contained herein have not been undertaken or done by unspecified sources or persons.

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ABSTRACT

The purpose of this project is to study the viscosity of synthesized polyol ester from jatropha oil. Jatropha oil-based polyol esters are potential biodegradable stocks for environmentally friendly lubricants. The physical properties of lubricants are primarily contributed by the structure of the lubricant base stock. Similarly, the first criterion for any potential polyol ester hydraulic fluid is the viscosity of the base ester before addition of any additive. Viscosity, μ, is the most important characteristic of a fluid lubricant because it has a major role in the formation of a fluid film. Hydraulic fluids of any type are designed to meet a specific viscosity classification. Viscosity grades are defined by International Standards Organization (ISO) standards according to a fluid's viscosity at 40ºC in centistokes (mm^2/second). The synthesis of jatropha oil trimethylolpropane (TMP) esters has been performed by transesterification of jatropha oil methyl esters (ME) with TMP using sulphuric acid as catalyst. The temperature and shear rate effect on viscosity are then have been investigated and be compared with methyl ester, sarapar and neopentylglycol in this project. The Brookfield Cap 2000+ L-Series and H-series have been used to measure viscosity at lower and higher temperature respectively. Polyol esters (POE) are being used in many applications areas as compressor oils, refrigeration lubricants, metalworking fluid, jet engine lubricants, high-temperature chain oils, hydraulic fluids and automotive gear oils. In industrial markets, POEs are used extensively in synthetic refrigeration lubricants due to their miscibility with non-chlorine refrigerants. They are also widely used in a variety of very high temperature applications such as industrial oven chains, stationary turbine engines, high temperature grease, fire resistant transformer coolants, fire resistant hydraulic fluids, and textile lubricants.

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ABBREVIATIONS AND NOMENCLATURE

TMP	trimethylpropane
ME	methyl ester
PAO	polyalphaolefin
PAG	polyalkylene glycols
FAME	fatty acid methyl ester
NPG	neopentyl glycol ester
RPM	revolution per minute
VI	viscosity index
Hz	hertz
Pa	Pascal
cSt	centistokes
v	kinematic viscosity
u	simple shear flow
τ	shear stress
F	force applied to top plate
R	cone radius
Θ_0	cone angle
Ω	radial distance from the axis of rotation

CHAPTER 1

INTRODUCTION

1.1 BACKGROUND OF STUDY

New technologies focused on the development of products from renewable sources have become very important during the last decade. This is due to increased concerns over the use of petroleum-based products caused by the progressive depletion of the world reserves of fossil fuels and concerns on their environmental impact. The global drive towards the use of biodegradable products is increasing despite early commercial resistance due to their cost and performance consideration.

In this project, the chemical synthesis of Jatropha oil trimethylpropane esters will be conducted via transesterification of Jatropha oil methyl esters with trimethylpropane (TMP) and the viscosity will be studied and analyzed. Depending on the types of multifunctional alcohol and monofunctional acid precursors, a wide variety of synthetic polyol esters can be obtained. The viscosity of mineral and synthetic oils significantly decreases when their temperature is raised. The higher viscosity is restored after the oils cool down to their original temperature.

The viscosity of synthetic oils is relatively less sensitive to temperature in comparison to mineral oils. But the viscosity of synthetic oils also decreases with increasing temperature. Development of non-edible oil production can decrease the risk of food security. Jatropha *(Jatropha curcas)* is one of such non-edible vegetable oils which have great production potential. It can be grown in very poor soils or idle lands and it has seeds with 30% to 40% approximate oil content. The Jatropha oil may be the key to addressing the problems of energy and food self-sufficiency. Cultivating the Jatropha oil does not only provide oil but can also ensure that agricultural lands devoted to food production will not be diverted to fuel crops.

1.2 PROBLEM STATEMENT

1.2.1 Problem Identification

Detailed knowledge of the viscometric properties of natural and chemically modified polyol ester is of particular importance whenever lubrication is their intended usage. The most important mechanical property of lubricant fluids is viscosity. For hydrodynamic lubrication, the viscosity, μ, is the most important characteristic of a fluid lubricant because it has a major role in the formation of a fluid film [1].

As example related to the effect of temperature towards lubricant viscosity, one of the problems in bearing design is the difficulty of precisely predicting the final temperature distribution and lubricant viscosity in the fluid film of the bearing [1]. For a highly loaded bearing combined with low speed, oils of relatively high viscosity are applied. However for high speed bearings, oils of relatively low viscosity are usually applied.

Minimum viscosity is required to secure proper hydrodynamic lubrication when the engine is at an elevated temperature. For this purpose, a lubricant of high viscosity at ambient temperature must be selected. This must result in high viscosity during starting of a car engine, particularly on cold winter mornings, causing heavy demand on the engine starter and battery [1]. For this reason, lubricants with less sensitive viscosity to temperature variations would have a distinct advantage. This brings to the introduction of synthetic oils that is also less sensitive to temperature variations, in comparison to regular mineral oils.

The physical properties of lubricants mainly contributed to the structure of the lubricant stock. Similarly, the first criterion for any potential polyol ester hydraulic fluid is the viscosity of the base ester before addition of any additive. Viscosity, μ, is the most important characteristic of a fluid lubricant because it has a major role in the formation of a fluid film [2].

1.2.2 Significant of Project

Structure of the lubricant base stock contributes to the physical properties of lubricants especially viscosity. Viscosity, μ, of the main ester is the most significant characteristic for any potential ester hydraulic fluid before addition of any additive [1]. Therefore, this project is very significant to investigate the viscosity of chemically synthesized polyol ester as the base stock for lubricant fluid.

The influence of temperature towards viscosity will help to predict the final temperature distribution and lubricant viscosity in the fluid film as example in the bearing operation. The problem when a lubricant is subject to vey large variations of viscosity due to changes in temperature will be solved. The viscosity of synthetic oils is less sensitive to temperature variation to be compare with mineral oil based lubricant and this would have a distinct advantage [3].

There are so many factors that cause change in viscosity like temperature, pressure and rate of shear and age of lubricant. If we are working with oils, it is necessary to know how the viscosity varies with the temperature. Correct determination of viscosity at any temperature is essential for designers and users when predicting the performance of lubricants [4].

Furthermore, Jatropha oil TMP esters are the potential biodegradable base stock for lubricant production that get an attention in recent years that has been paid to natural triglycerides, fatty acid derived from them and their potential [5]. To compete economically with petroleum-based lubricant, lubricant production cost can be reduced using less expensive vegetable oils including the non-edible Jatropha oil that may be the key to addressing the problems of energy and food self-sufficiency to compare with other edible vegetable oil as example rapeseed, soybean, palm oils and corn [6].

1.3 OBJECTIVES AND SCOPE OF STUDY

1.3.1 Objectives

1) To synthesize polyol ester by transesterfication of Jatropha oil methyl esters (ME) with trimethylolpropane (TMP).

2) To investigate the influence of temperature and shear rate towards the kinematic viscosity of synthesized polyol esters from jatropha oil and to compare it with methyl ester, sarapar and neopentyl glycol ester.

1.3.2 Scope of Study

This project is an experimental based study and can be divided into two parts throughout the given two semester's period. The first part of the project will be the preparation of polyol ester from Jatropha oil by transesterfication of Jatropha oil methyl esters (ME) with trimethylolpropane (TMP). A two step transesterification reactions will be performed in a batch process to convert Jatropha oil to its methyl ester with 1% sodium hydroxide (KOH) as catalyst and later on react it with TMP to produce polyol ester. The later part of this project will be the viscosity studies of the chemically synthesized polyol ester from Jatropha oil by comparing the viscosity at varies temperature and shear rate with methyl ester, sarapar and neopentyl glycol ester.

1.3.3 The Relevancy of Project

This project is relevant by looking into the importance of the biodegradable polyol ester as the base stock of lubricant production. Ester based synthetic lubricant are composed by several compounds. What is needed in completing this project is not only the understanding of how biodegradable polyol ester is synthesized by using transesterification process but also require the ability to investigate and analyse the importance of viscosity of polyol ester as the most important physical properties of the base stock in lubricant production.

This project addresses the importance of viscosity in fluid flow. Physical systems and applications as diverse as fluid flow in pipes, the flow of blood, lubrication of engine parts, the dynamics of raindrops, volcanic eruptions, planetary and stellar magnetic field generation, to name just a few, all involve fluid flow and are controlled to some degree by fluid viscosity. Therefore this project is relevance in the sense of it discuss the importance of viscosity as the most important physical property of lubricant.

In addition, this project address the importance of using Jatropha oil as the source of natural triglycerides and fatty acid to gradually replace the hydrocarbon-based lubricants derived from petroleum. This raw material is cheap and renewable. Moreover, natural fats and products derived from them are generally environmentally friendly. Synthetic esters produced from natural acids cannot be used at extremely high temperatures, but they are very suitable in less extreme applications such as two-stroke engine oils, chain bar oils, cutting oils, concrete mould release agents and cosmetic ingredients.

CHAPTER 2

THEORY

2.1 ANALYSIS OF LITERATURE

The main purpose of lubricants is decreased friction and wear, and other effects including heat transfer, contaminant suspension, liquid sealing and corrosion protection. Between 5000 to 10000 different lubricant formulations are necessary to satisfy more than 90% of all lubricant applications.

Polyol esters improve the performance of lubricants, whether used as the primary base stock or as a property-enhancing additive. Polyol esters can be produced over a wide range of viscosities, viscosity indices, load-carrying and other critical properties. The inherent polarity of esters results in lower volatility, cleaner lubrication, long-life, biodegradability and attraction to metal surfaces [7].

Polyol esters are formed by reacting an alcohol with two or more reactive hydroxyl groups. These fluids are used primarily for aircraft engines, high temperature gas turbines, hydraulic fluids and heat exchange fluids. Polyol esters are much more expensive than diesters [7]. Lubricating greases with polyol esters as the base fluid are particularly suited to high temperature applications.

Polyol esters have the same advantages/disadvantages as diesters. They are, however, much more stable and tend to be used instead of diesters where temperature stability is important. In general, a polyol ester is thought to be 40-50 ºC more thermally stable than a diester of the same viscosity. Esters give much lower coefficients of friction than those of polyalphaolefin (PAO) and mineral oil [8]. By adding 5-10% of an ester to a PAO or mineral oil the oil’s coefficient of friction can be reduced markedly [7].

Most lubricants, including mineral and synthetic oils, demonstrate a linear relationship between the shear stress and the shear rate [1]. A similar linear relationship holds in the air that is used in air bearings. Fluids that demonstrate such linear relationships are referred to as Newtonian fluids. In simple shear flow, u = u(y), the shear stress, τ, is proportional to the shear rate. The shear rate between two parallel plates without a pressure gradient is U=h. But in the general case of simple shear flow, u = u(y), the local shear rate is determined by the velocity gradient du/dy, where u is the fluid velocity component in the x-direction and the gradient du/dy is in respect to y in the normal direction to the sliding layers [9].

Some of the advantages of the synthetic lubricant by using chemically synthesized polyol ester as base stock are measurably better low and high temperature, viscosity performance, better chemical & shear stability, decreased evaporative loss, resistance to oxidation, thermal breakdown and oil sludge problems, extended drain intervals with the environmental benefit of less oil waste, improved fuel economy in certain engine configurations, better lubrication on cold starts and longer engine life [10].

Polyol esters can extend the high temperature operating range of a lubricant by as much as 50 - 100°C due to their superior stability and low volatility. They are also renowned for their film strength and increased lubricity which is useful in reducing energy consumption in many applications [11].

Maximum and minimum operating temperatures, along with the system's load, determine the fluid's viscosity requirements. For hydraulic fluid, the fluid must maintain a minimum viscosity at the highest operating temperature. However, the hydraulic fluid must not be so viscous at low temperature that it cannot be pumped.

2.2 VISCOSITY UNITS

The SI unit of pressure, *p*, as well as shear of stress, τ, is the Pascal (Pa) = Newtons per square meter [N=m2]. This is a small unit; a larger unit is the kilopascal (kPa) = 103 Pa. In the imperial (English) unit system, the common unit of pressure, *p*, as well as of shear stress, τ, is lbf per square inch (psi). The conversion factors for pressure and stress are:

$$\begin{aligned} 1 \text{ Pa} &= 1{:}4504 \times 10^{-4} \text{ psi} \\ 1 \text{ kPa} &= 1{:}4504 \times 10^{-1} \text{ psi} \end{aligned} \qquad (1)$$

The SI units of kinematic viscosity, *v*, are [m^2/s]. An additional cgs unit for absolute viscosity, m, is the poise [dyne-s/cm^2]. The unit of dyne-seconds per square centimeter is the poise, while the centipoise (one hundredth of poise) has been widely used in bearing calculations, but now has been gradually replaced by SI units. The cgs unit for kinematic viscosity, n, is the stokes (St) [cm2/s]. A smaller unit is the centistokes (cSt), cSt = 10^{-2} stokes. The unit cSt is equivalent to [mm2/s][12].

2.3 BIOLUBRICANTS

The primary functions of lubricants are decreased friction and wear, and other effects including heat transfer; contaminant suspension; liquid sealing and corrosion protection. Between 5,000-10,000 different lubricant formulations are necessary to satisfy more than 90% of all lubricant applications [13].

To meet these requirements most modern lubricants are complex formulated products consisting of 70-90% base oils mixed with functional additives to modify the natural properties (i.e. cold stability, oxidation stability, hydrolytic stability, viscosity and viscosity index, corrosion) and fulfil the requirements for the fluid.

Biolubricants are often, but not necessarily, based on vegetable oils. In all different current discussions biolubricants are at the least products for which formulation for rapid biodegradability and low environmental toxicity is a deliberate and primary intention. However, no universal agreement exists on the origin and chemical composition of the biolubricants. They can also be synthetic esters which may be partly derived from renewable resources i.e. the hydrolysis of fats and oils to produce the constituent fatty acids. They can be made from a wider variety of natural sources including solid fats and low grade or waste materials such as tallows.

The base oil can be mineral, vegetable, synthetic or re-refined:-

1) ·**Mineral oils** are the most commonly used today. They consist predominantly of hydrocarbons but also contain some sulphur and nitrogen compounds with traces of a number of metals.

2) **Vegetable oils** used in lubricants are mainly derived from rapeseed, sunflower, palm and coconut.

3) **Synthetic oils** include among others polyalphaolefins (PAO), synthetic esters and polyalkylene glycols (PAGs). PAOs are petrochemical derived synthetic oils that most resemble mineral oils. Synthetic esters form a large group of products, which can be either from petrochemical or oleochemical origin. Five different categories of synthetic esters can be distinguished: monoesters, di-esters, phthalate esters, polyol esters and complex esters.

4) **Re-refined oil** is used oil that undergoes an extensive rerefining process to remove contaminants to produce fresh base oil. The base oil is then sold to blenders who add additive packages to produce lubricants.

Genetically-modified vegetable oils, such as high-oleic sunflower and rapeseed, are also beginning to find use in applications where higher oxidative stability is needed [14]. Vegetable oils offer biodegradability and low toxicity. Obviously during the formulation of a biodegradable and low toxicity fluid the additives must be biodegradable and have low toxicity. In the beginning of the development of biolubricants (some 20 years ago) the focus was on the formulation of products based on pure vegetable oils. Over the last ten years, however, the trend has been the use of synthetic ester types which may be partly derived from renewable resources.

To avoid controversies associated with the term biolubricants, as well as to create a starting point for more environmental preferable products to enter the market, a European harmonization of the term is highly desirable. The two most significant opportunities for biolubricants are:

1) **High-risk lubricants** i.e. in applications where there is a high probability of accidental exposure of the lubricants to sensitive environments e.g. hydraulic equipment in forests and by water
2) **Total-loss lubricants –** where, by the design of the equipment or application the lubricant ends up almost entirely in the environment.

There is a necessity to develop more analytical tailor-made methods to better understand the nature of lubricants and to better study which are the modifications occurring during their use. Simply transferring the well-known methodologies from mineral oils to vegetable oils would result in an unrealistic picture of these products [15]. A preliminary chemical evaluation of base esters should detect defects that may cause problems during in-use operation. A strong effort must be made to set up and standardize methods for quality control.

2.4 BENEFITS OF BIODEGRADABLE LUBRICANTS

1) Less emissions because of higher boiling temperatures ranges of esters (Native triglycerides lead to partly gummy structures at high temperatures and can build eye irritant acroleins)[16]

2) Total free of aromatics, over, 90 % biodegradable oils, non water polluting, oil mist and oil vapor reduction, leading to less inhalation of oil mist into the lung

3) Better skin compatibility, less dermatological problems.

4) High cleanliness at the working place

5) At least equal and often higher tool life, because higher wetting tendency of polar esters lead to friction reduction.

6) Higher viscosity index, their viscosity does not vary with temperature as much as mineral oil. This can be an advantage when designing lubricants for use over a wide temperature range. This can also result in lower viscosity classes for the same applications combined with easier heat transfer [17].

7) Higher safety on a shop floor, because of higher flashpoints at the same viscosities

8) Cost savings on account of less maintenance, man power, storage and disposal costs.

2.5 DISADVANTAGES OF BIODEGRADABLE LUBRICANTS

1) Low temperature limitations. The addition of cosolvents such as synthetic fluids or mineral oils can improve the low-temperature properties of vegetable oils

2) Partly worse smell and compatibility with paintings and sealings

3) More flushing tendency because of lower viscosity

4) Less oxidation and hydrolytic stability's of vegetable oils

5) Filter clogging tendency depending on ester type and formulation

CHAPTER 3

METHODOLOGY

3.1 CHEMICALS

3.1.1 Jatropha Oil

Approximately 100 g of *Jatropha curcas* oil that can readily be obtained from laboratory will be used to react it with methanol to obtain the fatty acid methyl ester (FAME) in the two steps transesterification process.

3.1.2 Methanol

Excess methanol is needed in the experiment with the molar ratio to oil is 6:1.

3.1.3 Sodium Hydroxide (KOH)

Sodium hydroxide employs as a catalyst in this transesterification process. 1 % KOH will be used during the experiment.

3.1.4 Sulphuric Acid

Sulphuric acid employs as a catalyst in this transesterification process. 1 % H_2SO_4 will be used during the experiment.

3.1.5 Trimethylpropane (TMP)

TMP that can readily be obtained from laboratory will be reacted with FAME producing desired polyol ester and also methanol.

3.1.6 Sarapar

Synthetic base drilling fluid, chemical intermediate. Purchased and to be used in comparison of the viscosity.

3.1.7 Neopentyl Glycol (NPG) ester

Product of esterification neopentyl glycol ester with hexanoic acid. Purchased and to be used in comparison of the viscosity.

3.2 PROJECT ACTIVITIES

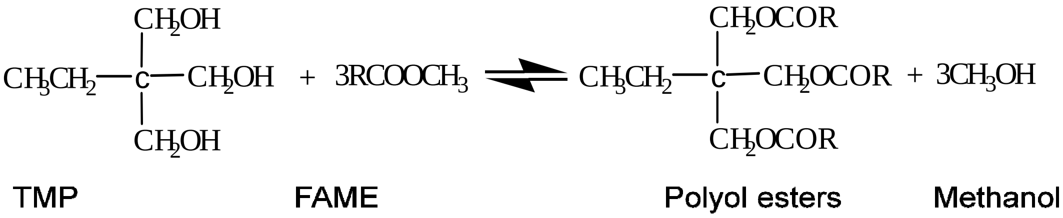
3.2.1 Synthesis of Polyol Esters from Jatropha Oil

Transesterification reactions were to perform in a batch process. A two-step transesterification process will be used because of the high free fatty acid content of Jatropha oil which causes fatty acid salts (soap) formation during base-catalyzed transesterification. The soap could prevent separation of the methyl ester layer from the glycerine fraction. Conditions of the reaction are the following:

1) Methyl ester/TMP ratio: 4:1
2) Temperature : 210 ºC
3) Pressure : 0.2-20mbar
4) Reaction time : less than 1 hour
5) Catalyst : Sulfuric acid 1%

The first step is acid esterification and pre-treatment for removing free fatty acids in the oil, which is mainly a pre-treatment process. In this step, free fatty acids were first converted to esters with methanol using an acid catalyst H_2SO_4 . The reactor will be filled about approximately 100g Jatropha oil. The catalyst, 1% sulphuric acid is dissolved in methanol and then added to the reactor at a reaction temperature of 70ºC. The mixture will be agitated throughout the experiment at 700 rpm. After about one hour of reaction, the mixture will be allowed to settle and the methanol-water friction at the top layer will be removed.In the second step, the product of the pretreatment step will be poured into the reactor and heated at 50ºC. The catalyst, sodium hydroxide (1 %) is dissolved in methanol and then added to the reactor. The reaction was carried out using 100% excess methanol and the molar ratio of methanol to oil is 6:1. After the end of the reaction (1.5h), the mixture will be cooled to room temperature and transferred to a separatory funnel. The two layers were separated by sedimentation. The methyl ester phase will be washed with hot distilled water[18].

(2)



$$CH_3CH_2-\overset{CH_2OH}{\underset{CH_2OH}{C}}-CH_2OH + 3RCOOCH_3 \rightleftharpoons CH_3CH_2-\overset{CH_2OCOR}{\underset{CH_2OCOR}{C}}-CH_2OCOR + 3CH_3OH$$

Methyl ester will then allowed to react with trimethylpropane (TMP) at temperature 210 ºC with the presence of sulphuric acid 1% catalyst with the molar ratio FAME : TMP 4 : 1. When the reaction is complete, the synthesized ester will be separated from the catalyst by using filtration.

3.2.2 Viscosity Studies of Synthesized Polyol Esters from Jatropha Oil

Brookfield digital viscometer model CAP 2000+ L-Series and H-Series have been used to measure the viscosity of the sample at different temperature and shear rate. L-Series viscometer is used to measure viscosity and shear stress at lower temperature in the range of 5ºC to 60 ºC meanwhile H-Series viscometer is used to measure viscosity and shear stress at higher temperature in the range of 65ºC to 120 ºC.

Operating environment : room temperature
Viscosity accuracy : ±1% full scale range in use
Viscosity repeatability : 0.2% of full scale range in use
Temperature accuracy : ±1ºC: -100ºC to 149ºC, ±2ºC: 150ºC to 300ºC
Spindle number : 2

Figure 3.1: Brookfield Viscometer CAP 2000+ L-Series

Experiment is started by measuring the viscosity of polyol ester at different temperature range of 5 ºC to 120 ºC and later on proceeded with measuring the shear stress at different shear rate range of 100 rpm to 1000rpm. Spindle number 2 is chosen and the methodologies are later on proceed with three other different samples that are methyl ester, sarapar and neopentyl glycol at specific temperature considering the boiling point for each of the samples. Details are below:-

Table 3.1: Sample Chosen Temperature and Shear Rate Range

Sample	Chosen Temperature Range (ºC)	Shear Rate Range (rpm)
Polyol ester	5 - 120	100 - 1000
Methyl ester	5 - 120	100 - 1000
Neopentyl glycol	5 - 120	100 - 1000
Sarapar	5 – 55	100 - 1000

For temperature study, viscosity data (in centipoise) are measured for every 5 ºC increasing temperature. Details of the methodology for measuring viscosity by using Brookfield viscometer CAP 2000+ are as per below:-

1) Cone spindle is put in the down position and to make sure the sample trap is covering the spindle.
2) The viscometer is allowed to stabilize for at least 30 minutes at 25ºC (low temperature for L-Series) or at 60ºC (high temperature for H-Series).
3) At the end of the temperature stabilization period, 2 ml of sample is dispensed on the plate.
4) The viscosity test is run and the value is recorded when the reading is stabilized.
5) The test is repeated again for three times to obtain a more accurate value and result.

For shear stress study, shear rate data are measured at temperature 5 ºC and 60 ºC. Details of the methodology for measuring shear stress by using Brookfield viscometer CAP 2000+ are as per below:-

1) Cone spindle is put in the down position and to make sure the sample trap is covering the spindle.
2) The viscometer is allowed to stabilize for at least 30 minutes at 25ºC (low temperature for L-Series) or at 60ºC (high temperature for H-Series).
3) At the end of the temperature stabilization period, 2 ml of sample is dispense on the plate.
4) The viscosity test is run and the value is recorded when the reading is stabilized.
5) Print button is pressed to print out the shear stress value that is not being displayed at viscometer.
6) The test is repeated again for three times to obtain a more accurate value and result.

3.2.3 CAP Viscometer

The Brookfield CAP series of Cone/Plate Viscometers offer high shear rates and variable speeds in an instrument optimized for R&D and QC applications such as paints, coatings, resins, inks, cosmetics, pharmaceuticals and foods. These series of viscometers offer high shear rate with integrated temperature control for test sample volume of less

than 1 mL. The CAP series operates with automatic cone gap positioning and viscosity range calibration and is offered as two models: CAP 1000 and CAP 2000. The CAP 1000 is a single speed viscometer and has a fixed shear rate at 750 RPM on 50 Hz and 9000 RPM on 60 Hz and generates shear rates at 12,000 or 3,000 sec-1 at 60 Hz and 10,000 or 2,500 sec-1 at 50 Hz. Viscosity ranges from .25 to 100 Poise (0.25 to 10 Pa•s) depending on the cone spindle used. In this project, viscometer CAP 2000 is used. Tthe CAP 2000 is a variable-speed instrument and has variable shear rate capability over the speed range from 50 to 2,000 RPM. This instrument generates shear rates from 166 to 26, 600 sec-1 at viscosity ranges from 0.1 to 1,500 Poise (0.1 to 150 Pa•s). Both the CAP 1000 and CAP 2000 are accurate to ±2% of the full scale range and meet industry test standards BS3900, ISO 2884, and ASTM D-4287.

3.2.4 Viscosity Measurement

The simple cone-and-plate viscometer geometry provides a uniform rate of shear and direct measurements of the first normal stress difference. It is the most popular instrument for measurement of non-Newtonian fluid properties [19]. The working shear stress and shear strain rate equations can be easily derived in spherical coordinates, as indicated by the geometry in Figure 3.3.

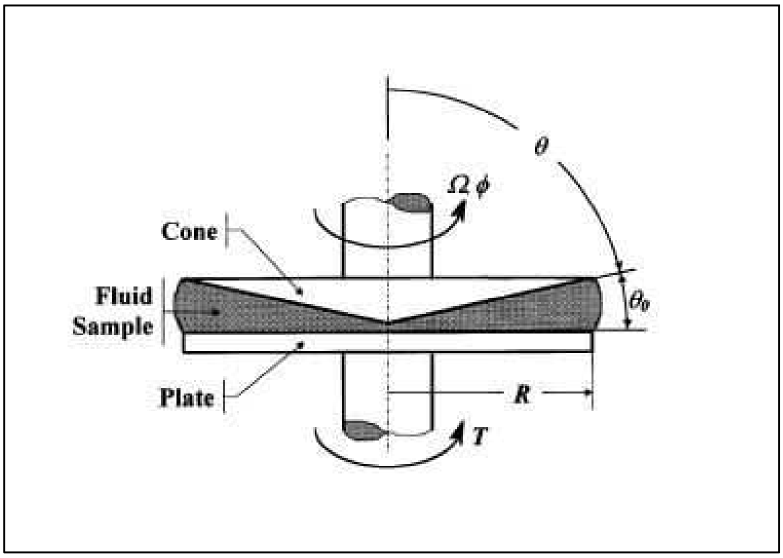


Figure 3.2: Cone and Plate Viscometer Geometry [9]

Viscosity is defined as the internal friction of a fluid. The microscopic nature of internal friction in a fluid is analogous to the macroscopic concept of mechanical friction in the system of an object moving on a stationary planar surface. Energy must be supplied to overcome the inertial state of the interlocked object and plane caused by surface roughness, and to initiate and sustain motion of the object over the plane. In a fluid, energy must be supplied to create viscous flow units by breaking bonds between atoms and molecules, and to cause the flow units to move relative to one another.

The resistance of a fluid to the creation and motion of flow units is due to the viscosity of the fluid, which only manifests itself when motion in the fluid is set up. Since viscosity involves the transport of mass with a certain velocity, the viscous response is called a momentum transport process. The velocity of flow units within the fluid will vary, depending on location. Consider a liquid between two closely spaced parallel plates as shown in Figure 3.3 A force, F, applied to the top plate causes the fluid adjacent to the upper plate to be dragged in the direction of F.

3.3 Gantt Chart/Milestone for the Second Semester of 2-Semester Final Year Project

No.	Detail/Week	1	2	3	4	5	6	7	8	9	10	11	12	13	14
1	Obtain sample	🟩	🟩												
2	Literature review	🟩	🟩	🟩	🟩	🟩	🟩	🟩	🟩	🟩	🟩	🟩	🟩	🟩	
3	Polyol ester studies			Δ											
4	Methyl ester studies				Δ										
5	Sarapar studies					Δ									
6	Neopentyl glycol studies						Δ								
7	Data interpretation			🟩	🟩	🟩	🟩	🟩	🟩	🟩	🟩	🟩	🟩	🟩	
8	Progress report 1	🟩	🟩	🟩	🟩	🟩 Δ									
9	Poster Presentation									Δ					
10	Dessertation	🟩	🟩	🟩	🟩	🟩	🟩	🟩	🟩	🟩	🟩	🟩	🟩	🟩	🟩 Δ
11	Final presentation														Δ

Δ	Milestone
🟩	Proces

CHAPTER 4

RESULTS & DISCUSSION

4.1 TEMPERATURE EFFECT ON VISCOSITY

Table 4.1: Effect of Temperature on the Viscosity of Polyol Ester

Temperature °C	**Viscosity 1**	**Viscosity 2**	**Viscosity 3**	**Viscosity, μ (cP)**
5	167	166	169	168.6
10	139	142	141	141.0
15	115	120	121	119.8
20	106	110	104	104.4
25	89	88	88	88.4
30	80	80	80	80.2
35	74	74	73	74.0
40	69	71	68	69.8
45	70	69	68	68.8
50	68	66	66	66.4
55	66	65	65	64.6
60	64	63	64	63.4
65	56	52	54	53.2
70	52	48	48	48.0
75	46	52	51	51.0
80	55	50	52	52.3
85	52	52	40	48.0
90	38	51	52	47.0
95	49	51	49	49.7
100	51	51	44	48.7
105	46	35	50	43.7
110	45	59	50	52.0
115	56	51	50	52.3
120	58	54	52	54.7

The viscosity of most fluids decreases with an increase in temperature. By measuring viscosity at two temperatures (using a single spindle and speed), it is possible to predict a flow curve representing the temperature dependence of the viscosity of a fluid. The experiments were carried out at temperature 5ºC to 120ºC without any additional of additives with the aim of observing the effect of temperature on polyol ester viscosity.

The effect of temperature on the viscosity of TMP and jatropha oil methyl ester at ambient pressure is shown in figure. As expected, the viscosity exhibits an exponential decrease with temperature, as illustrated by the data. The synthesized polyol ester could in principle be used as lubricant stocks at or above ambient temperature if only the viscosity were taken into account (and with the aid of antioxidant/anti hydrolysis/antirust packages) [21].

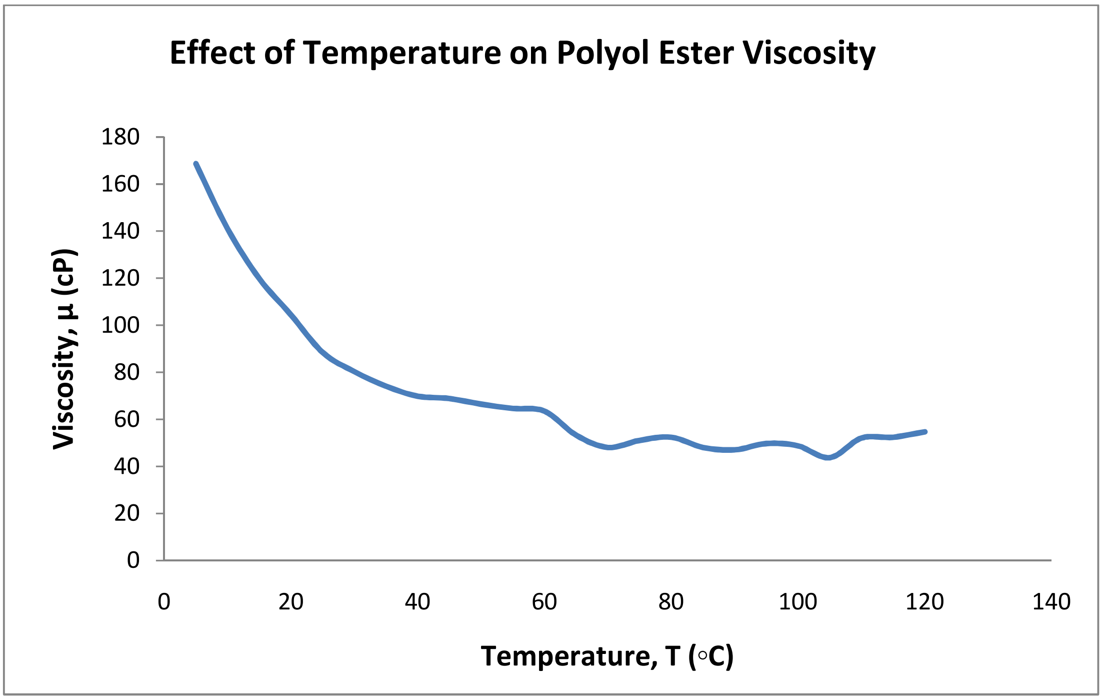
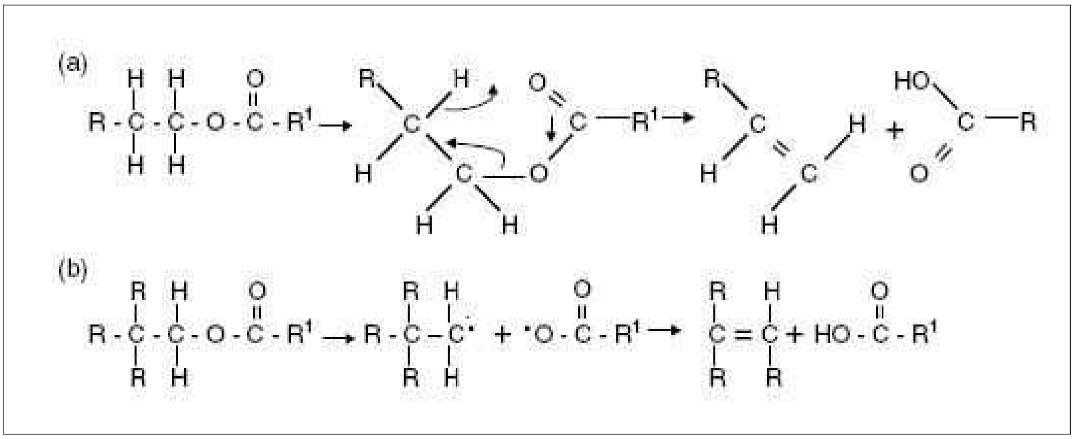


Figure 4.1: Viscosity vs. Temperature Plot for Polyol Ester

In Figure 4.1, it is observed that viscosity is decreasing exponentially and achieved a high stability at higher temperatures. The advantages in thermal stability of polyol esters are well documented and have been investigated on a number of occasions. It has been found that the absence of hydrogen atoms on the beta-carbon atom of the alcohol portion of the ester leads to superior thermal stabilities [20].

The presence of such hydrogen atoms enables a more favourable decomposition mechanism to operate via a six-membered cyclic intermediate producing acids and alkenes, (Figure [a]). When beta-hydrogen atoms are replaced by alkyl groups this mechanism cannot operate and decomposition occurs via a less favorable free-radical pathway. This type of decomposition requires more energy and can only occur at higher temperatures, (Figure [b]).

(3)



Hydraulic fluids of any type are designed to meet a specific viscosity classification. Viscosity grades are defined by ISO standards according to a fluid's viscosity at 40ºC in centistokes (mm^2/second). The first criterion for any potential new polyol ester hydraulic fluid is the viscosity of the base ester before any additives.

If the base ester viscosity needs to be increased to meet a specific ISO grade, polymer-based rheological modifiers are often added. Sometimes called thickeners, these additives not only increase a fluid's viscosity but also increase its viscosity index (VI). VI is a measure of a fluid's viscosity change as a function of temperature. Fluids with high VI are less affected by temperature and are referred to as "multigrade".

One drawback of using rheological modifiers is that they can be very difficult to dissolve in the base ester. A wide variety of polymers are available, but the selection of proper polymer and the fluid manufacturing process are difficult.

The desired outcome of research into new technologies was to design base esters that would meet standard ISO viscosity grades, have high viscosity index values. Table 4.2 shows the data for effect of temperature on the viscosity of methyl ester.

Table 4.2: Effect of Temperature on the Viscosity of Methyl Ester

Temperature °C	**Viscosity 1**	**Viscosity 2**	**Viscosity 3**	**Viscosity, μ (cP)**
5	75	72	78	75
10	67	60	60	62
15	50	58	54	54
20	52	46	53	50
25	46	47	47	47
30	45	49	43	46
35	44	47	46	46
40	41	48	45	45
45	45	39	47	44
50	37	45	43	42
55	39	41	39	40
60	40	39	46	42
65	36	43	34	38
70	38	42	37	39
75	40	37	36	38
80	38	41	36	38
85	35	39	36	37
90	38	37	34	36
95	33	38	35	35
100	37	31	35	35
105	38	34	30	34
110	31	37	29	33
115	31	29	35	32
120	28	32	29	30

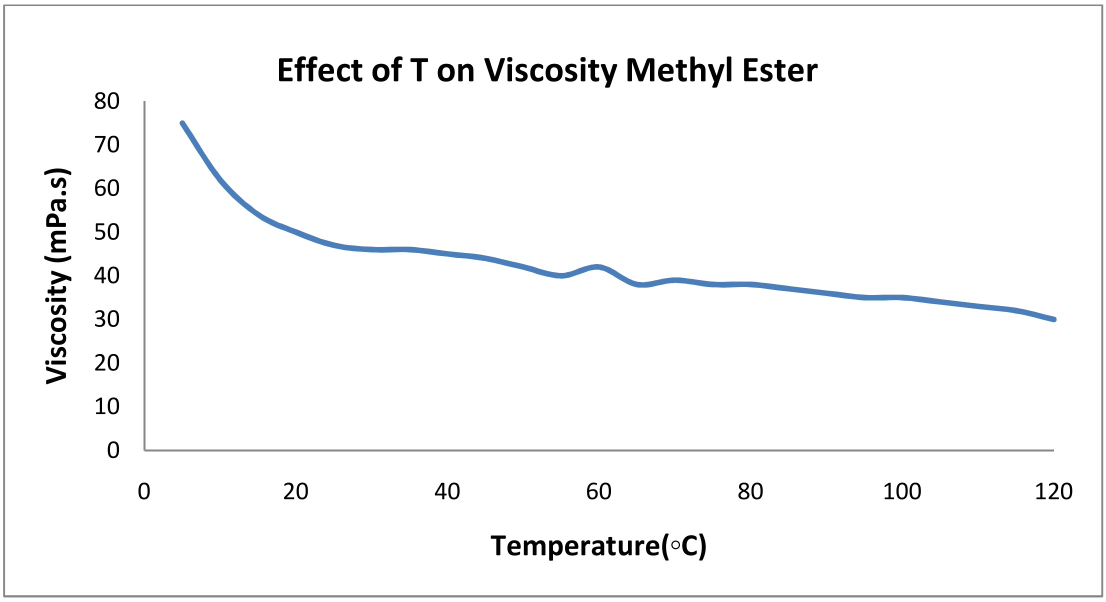


Figure 4.2: Viscosity vs. Temperature Plot for Methyl Ester

In particular, sarapar drilling base oil recorded a sharp increase in demand due to increased drilling activities by oil companies. This upward trend is expected to continue in the coming year with expected increase in oil and gas exploration and production drilling activities. Table 4.3 below shows the data for effect of temperature on the viscosity of methyl ester.

Table 4.3: Effect of Temperature on the Viscosity of Sarapar

Temperature °C	**Viscosity 1**	**Viscosity 2**	**Viscosity 3**	**Viscosity, μ (cP)**
10	56	61	55	58
15	55	59	56	57
20	58	53	53	56
25	50	56	58	55
30	49	55	48	52
35	49	55	47	51
40	54	50	47	51
45	46	52	53	51
50	45	49	55	50
55	48	46	51	49

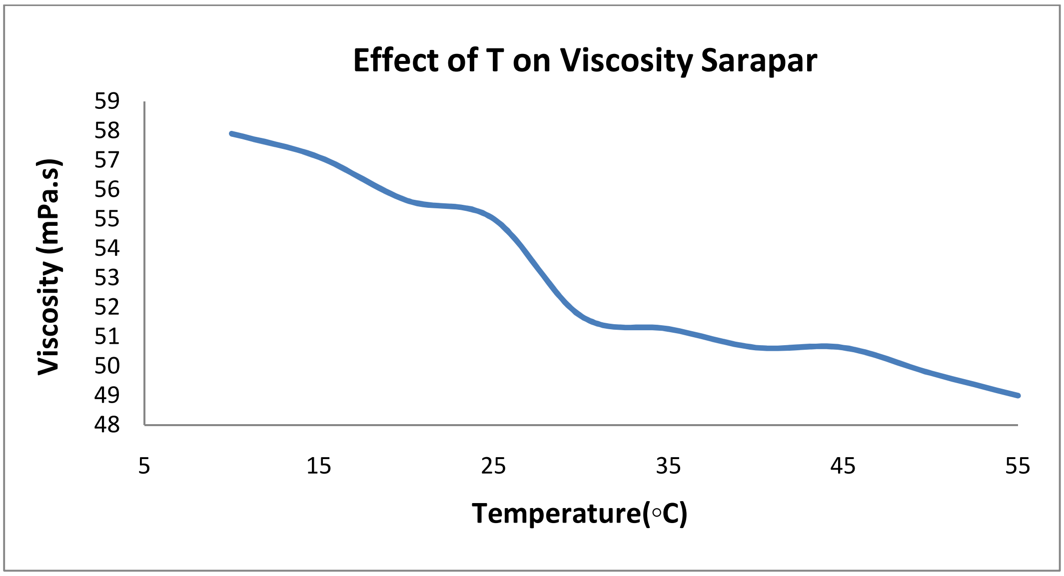


Figure 4.3: Viscosity vs. Temperature plot for Sarapar

Neopentyl polyol esters have not beta hydrogens in the formula, which are more hydrolytically and thermally stable than diesters. With excellent high and low temperature property, high flash point and low pour point, neopentyl polyol esters are suitable base oils for aviation gas turbine lubricant, high temperature chain oil, synthetic air compressor oil and refrigerator oil which is compatible with environmental protection requirement.

Neopentyl glycol is mainly used to produce unsaturated resin, polyester powder coating, oil-free alkyd resins, polyurethane foam plastic, that is, elastomer plasticizers, synthetic plasticizers,surfactants, insulation materials, printing inks, inhibitor and synthetic aviation lubricant oil additives. In addition, there are also applications in the pharmaceutical industry. Meanwhile, neopentyl glycol is also a good solvent, which can be used as selective separation for naphthenic base hydrocarbons. Table 4.4 below shows the data for effect of temperature on the viscosity of neopentyl glycol ester.

Table 4.4: Effect of Temperature on the Viscosity of Neopentylglycol Ester

Temperature °C	Viscosity 1	Viscosity 2	Viscosity 3	Viscosity, μ (cP)
5	75	72	78	75
10	67	60	60	62
15	50	58	54	54
20	52	46	53	50
25	46	47	47	47
30	45	49	43	46
35	44	47	46	46
40	41	48	45	45
45	45	39	47	44
50	37	45	43	42
55	39	41	39	40
60	40	39	46	42
65	36	43	34	38
70	38	42	37	39
75	40	37	36	38
80	38	41	36	38
85	35	39	36	37
90	38	37	34	36
95	33	38	35	35
100	37	31	35	35
105	38	34	30	34
110	31	37	29	33
115	31	29	35	32
120	28	32	29	30

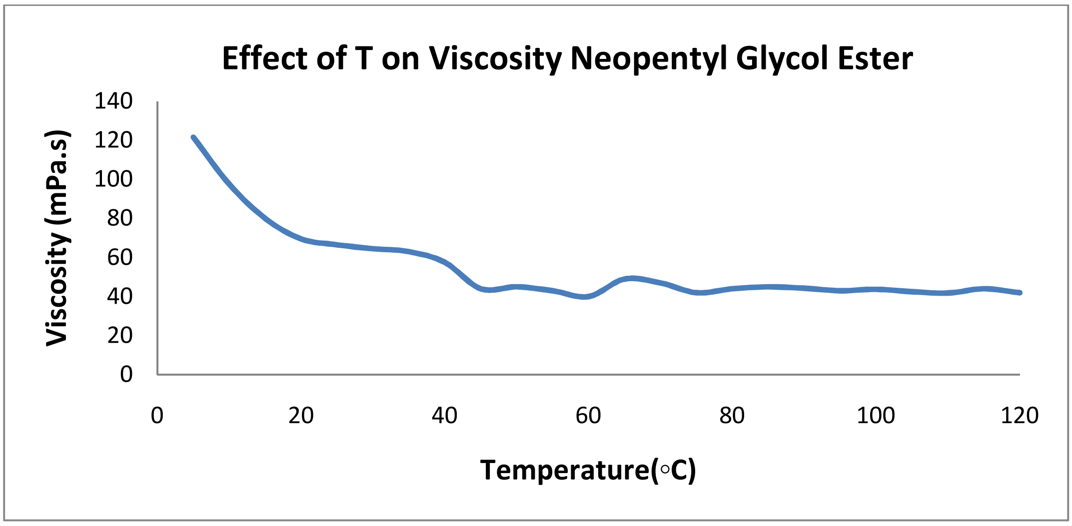


Figure 4.4: Viscosity vs. Temperature Plot for Neopentyl Glycol Ester

Figure 4.5 below shows the viscosity versus temperature comparison between methyl ester, polyol ester, sarapar and neopentyl-glycol ester. It is observed that polyol ester has the highest viscosity at all temperatures to be compared with other comparison fluids that are methyl ester, sarapar, and neopentyl-glycol ester. The bio lubricant obtained from jatropha oil presents a high viscosity when compared with the mineral lubricant oil that is sarapar because of the presence of small amount of triglyceride to be compared with sarapar. It was not possible to measure the viscosity of sarapar at higher temperature due to its boiling point that is at 62ºC.

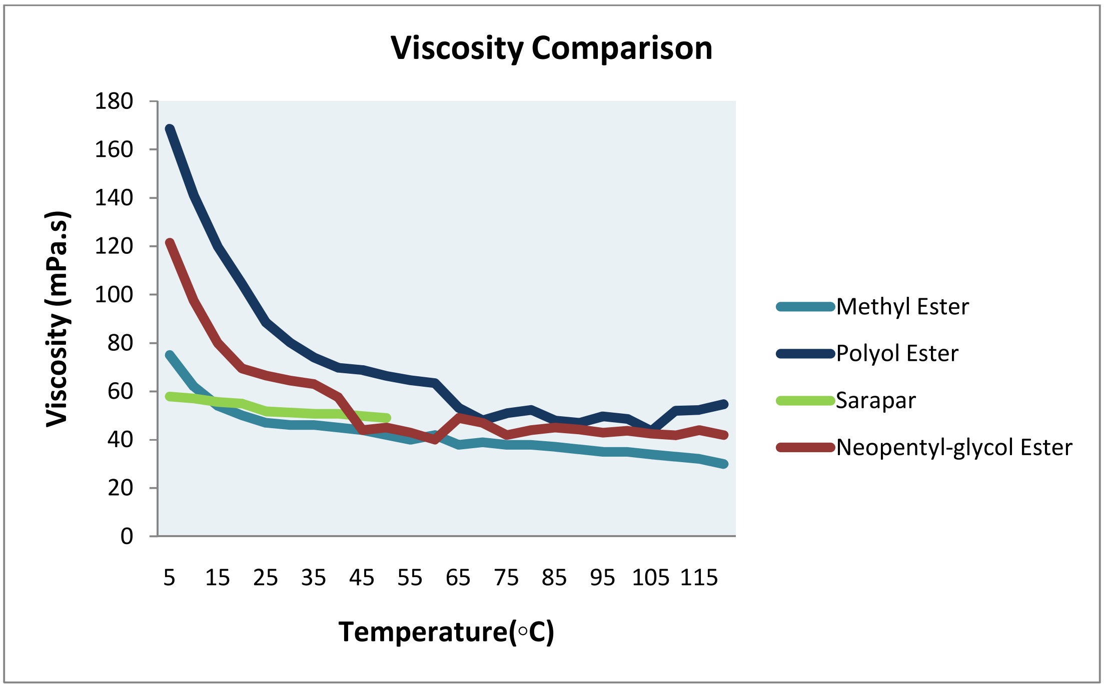


Figure 4.5: Viscosity vs. Temperature Comparison Between Methyl Ester, Polyol Ester, Sarapar and Neopentyl Glycol Ester

The ester linkage is an exceptionally stable one. Bond energy determinations predict that the ester linkage is more thermally stable than the C-C bond. Primarily, the thermal stability of ester lubricants are dependent on the presence of hydrogens on beta-carbons (avoided by use of polyol esters) and the number and type of hydrogens present. In decreasing order of stability –CH3 > -CH2- > -CH-. Generally therefore, linear acid esters are more stable than branched and short-chain acidesters are more stable than long. Stability of the alcohol used for polyol esters also determines the thermal stability in decreasing order of stability, PE > DiPE > TMP > NPG.

In this experiment, it is observed that polyol ester has the higher stability than neopentyl-glycol ester (NPG) especially at all temperatures because the generally, TMP esters of fatty acids have higher viscosity than that of analogous NPG esters, probably due to the fact that there are three acid groups in the structure of TMP esters and only two such groups in the molecules of NPG esters. Higher viscosity is very desirable property in view of their use as lubricants.

The viscosity of liquid with high viscosity indices changes very little with temperature. High viscosity is characteristic of compounds which have long, unbranched hydrocarbon chains as example fatty acids of most natural triglycerides. From the graph, it is clear that ester based on TMP are characterized by better thermal stability than NPG ester.

4.2 SHEAR RATE EFFECT ON VISCOSITY

Non-Newtonian fluids tend to be the rule rather than the exception in the real world, making an appreciation of the effects of shear rate a necessity for anyone engaged in the practical application of rheological data. It would, for example, be disastrous to try to pump a dilatant fluid through a system, only to have it go solid inside the pump, bringing the whole process to an abrupt halt. While this is an extreme example, the importance of shear rate effects should not be underestimated.

When a material is to be subjected to a variety of shear rates in processing or use, it is essential to know its viscosity at the projected shear rates. If these are not known, an estimate should be made. Viscosity measurements should then be made at shear rates as close as possible to the estimated values. It is frequently impossible to approximate projected shear rate values during measurement due to these values falling outside the shear rate range of the Viscometer. In this case, it is necessary to make measurements at several shear rates and extrapolate the data to the projected values. This is not the most accurate method for acquiring this information, but it is often the only alternative available, especially when the projected shear rates are very high. In fact, it is always advisable to make viscosity measurements at several shear rates to detect rheological behavior that may have an effect on processing or use. In lubrication application, the typical varying shear rate is between 10^3 to 10^7 as example in gasoline engines application. In this project the effect of shear rate towards the viscosity for polyol ester and the comparison fluids is investigated. Figure 4.7, 4.8, 4.9 and 4.10 shows the individual effect of shear rate meanwhile Figure 4.11 shows the graph for comparison of the shear stress data for all the comparison fluids.

Table 4.5: Effect of Shear Rate on the Shear Stress of Polyol Ester

Temperature °C	**Viscosity, µ (mPa.s)**	**Shear Rate (s^{-1})**	**Shear Stress (Pa)**
5	210	1333	280
	176	2667	469
	163	4000	652
	158	5333	840
	154	6667	1025
	151	8000	1210
	150	9333	1400
	150	10667	1600
	146	12000	1754
	146	13333	1940
60	73	1333	97
	57	2667	152
	52	4000	208
	54	5333	288
	53	6667	353
	53	8000	420
	46	9333	429
	40	10667	427
	35	12000	420
	34	13333	453

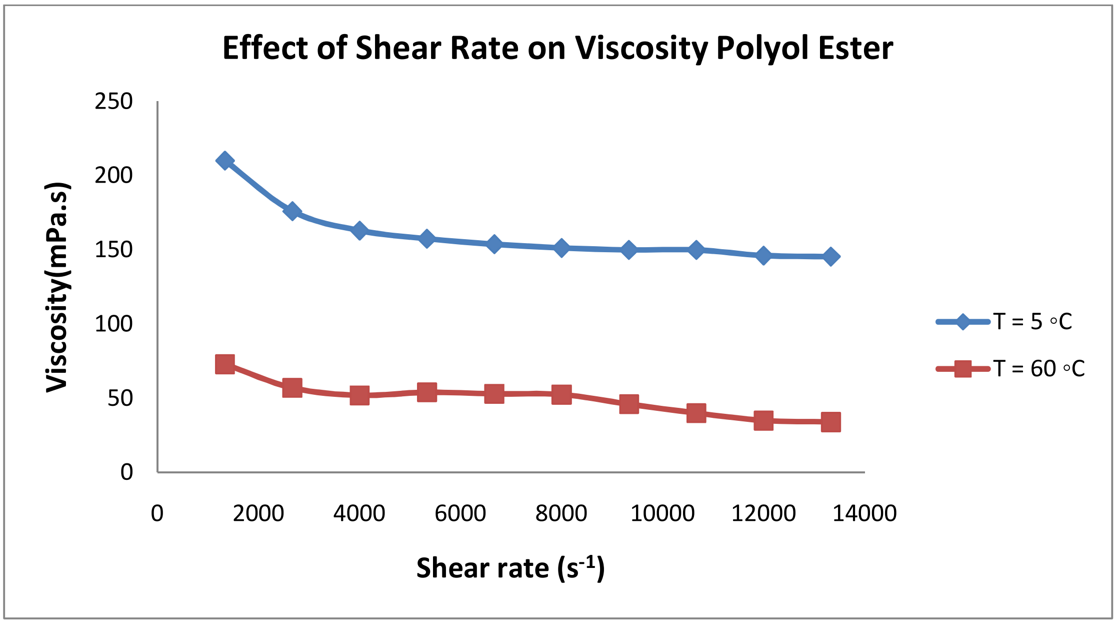


Figure 4.6: Comparison Effect of Shear Rate on Polyol Ester at Different Temperature

Table 4.6: Effect of Shear Rate on the Shear Stress of Methyl Ester

Temperature °C	Viscosity, µ (mPa.s)	Shear Rate (s^{-1})	Shear Stress (Pa)
5	65	1333	86
	56	2667	149
	53	4000	212
	53	5333	282
	45	6667	300
	48	8000	383
	39	9333	362
	40	10667	421
	46	12000	557
	43	13333	575
60	59	1333	79
	52	2667	139
	42	4000	168
	38	5333	203
	30	6667	200
	29	8000	232
	27	9333	252
	26	10667	277
	28	12000	330
	24	13333	325

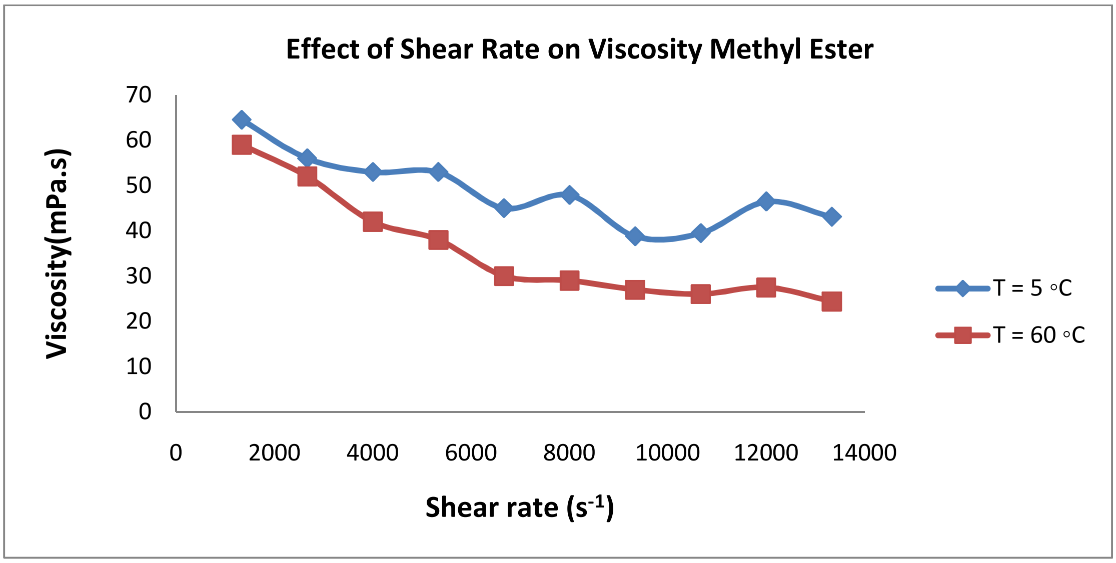


Figure 4.7: Comparison Effect of Shear Rate on Methyl Ester at Different Temperature

Table 4.7: Effect of Shear Rate on the Shear Stress of Sarapar

Temperature °C	Viscosity, µ (mPa.s)	Shear Rate (s^{-1})	Shear Stress (Pa)
10	60	1333	80
	59	2667	156
	58	4000	232
	56	5333	299
	55	6667	367
	51	8000	408
40	55	1333	73
	53	2667	141
	52	4000	207
	49	5333	260
	46	6667	307
	42	8000	337

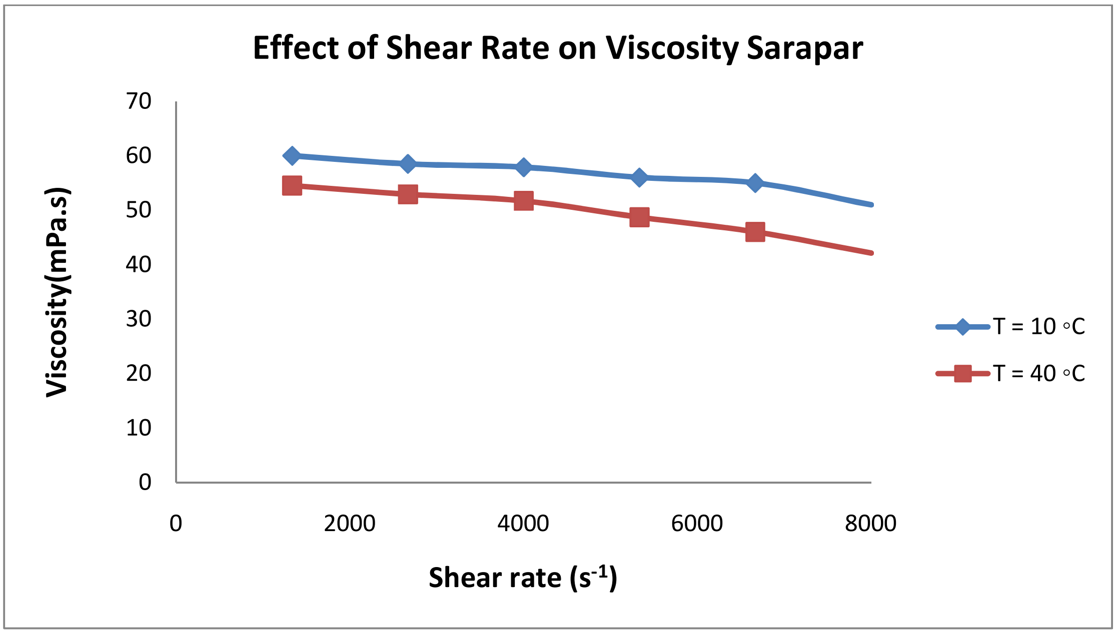


Figure 4.8: Comparison Effect of Shear Rate on Sarapar at Different Temperature

Table 4.8: Effect of Shear Rate on the Shear Stress of Neopentyl Glycol Ester

Temperature °C	**Viscosity, μ (mPa.s)**	**Shear Rate (s^{-1})**	**Shear Stress (Pa)**
5	65	1333	86
	57	2667	149
	55	4000	212
	54	5333	282
	55	6667	300
	57	8000	383
	55	9333	362
	48	10667	421
	44	12000	557
	40	13333	575
60	54	1333	87
	42	2667	151
	40	4000	220
	39	5333	289
	37	6667	366
	38	8000	452
	36	9333	515
	35	10667	512
	33	12000	528
	29	13333	533

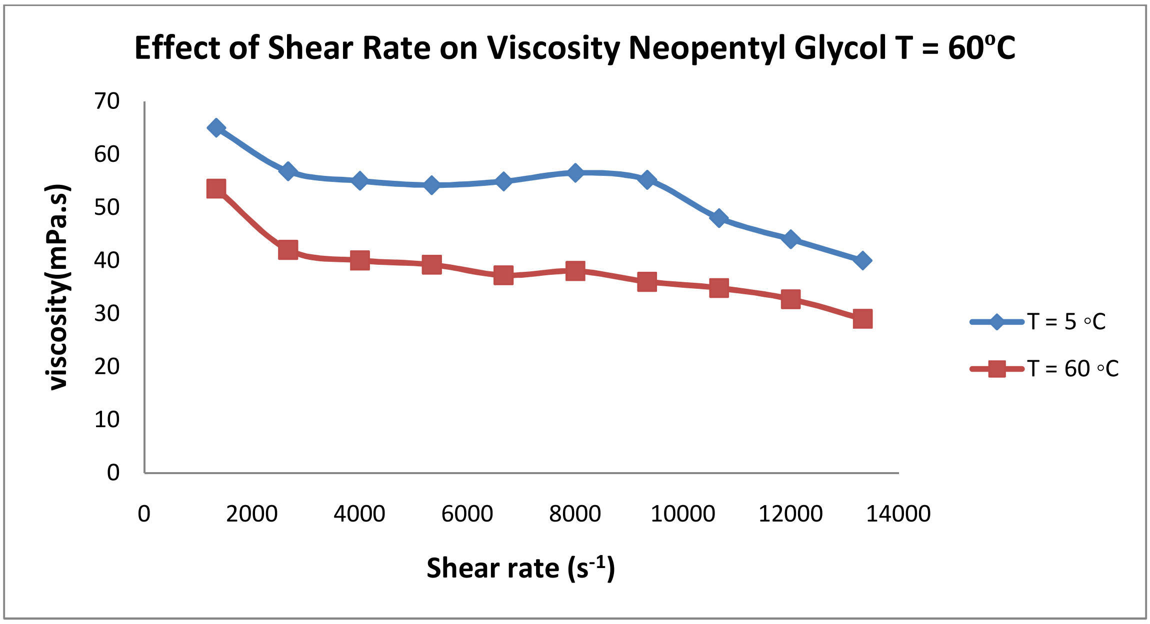


Figure 4.9: Comparison Effect of Shear Rate on Neopentyl Glycol Ester at Different Temperature

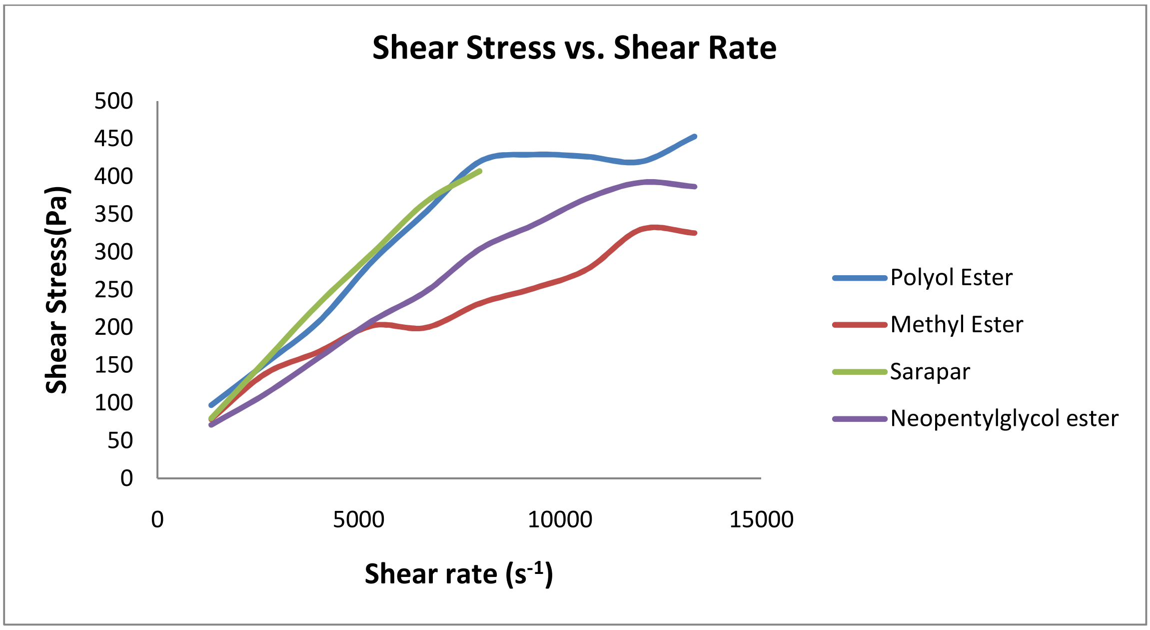


Figure 4.10: Shear Stress vs. Shear Rate at 60ºC

From the graph of shear stress versus shear rate of all the comparison oil above, it can be concluded that all of the liquid display a decreasing viscosity with an increasing shear rate. This type of flow behaviour is sometimes called “shear-thinning.” Shear rate is the rate at which adjacent layers of a fluid move with respect to each other, usually expressed as reciprocal seconds. When the fluid is placed between two parallel surfaces moving relative to each other, shear rate (s^{-1}) is equal to the relative velocity of surface (m/s) distance between surfaces.

Shear stress is the frictional force overcome in sliding one "layer" of fluid along another, as in any fluid flow. The shear stress of a petroleum oil or other Newtonian fluid at a given temperature varies directly with shear rate (velocity). The ratio between shear stress and shear rate is constant; this ratio is termed viscosity. The higher the viscosity of a Newtonian fluid, the greater the shear stress as a function of rate of shear. In a non-Newtonian fluid, shear stress is not proportional to the rate of shear. A non-Newtonian fluid may be said to have an apparent viscosity, a viscosity that holds only for the shear rate (and temperature) at which the viscosity is determined. The shear failure point in most oils is between 4,000 and 8,000 psi.

CHAPTER 5

CONCLUSION & RECOMMENDATION

5.1 CONCLUSION

Development of non-edible oil production can decrease the risk of food security. Jatropha *(Jatropha curcas)* is one of such non-edible vegetable oils which have great production potential. It can be grown in very poor soils or idle lands and it has seeds with 30% to 40% approximate oil content. The Jatropha oil may be the key to addressing the problems of energy and food self-sufficiency. Cultivating the Jatropha oil does not only provide oil but can also ensure that agricultural lands devoted to food production will not be diverted to fuel crops.

The viscosity of most fluids decreases with an increase in temperature. By measuring viscosity at two temperatures (using a single spindle and speed), it is possible to predict a flow curve representing the temperature dependence of the viscosity of a fluid. In this project, t is concluded that polyol ester has the highest viscosity at all temperatures to be compared with other comparison fluids that are methyl ester, sarapar, and neopentyl-glycol ester.

It is frequently impossible to approximate projected shear rate values during measurement due to these values falling outside the shear rate range of the viscometer. In this case, it is necessary to make measurements at several shear rates and extrapolate the data to the projected values. This is not the most accurate method for acquiring this information, but it is often the only alternative available, especially when the projected shear rates are very high. In fact, it is always advisable to make viscosity measurements at several shear rates to detect rheological behavior that may have an effect on processing or use.

5.2 RECOMMENDATION

The high price of biolubricants (both on the basis of vegetable oils and especially based on synthetic esters) is the main restriction for the development of biolubricants at the current time. They are generally between 1.5 and 5 times more expensive than conventional lubricants. Economical and environmental balance needs to be performed in order to minimise the higher price i.e. economic cost versus true cost. The market for natural esters is increasing, but nevertheless strong efforts are still needed from ester base oil, additive and lubricant industry to develop improved products which will be generally more accepted by toxicologists and legislation as well as by end users.

There is a necessity to develop more analytical tailor-made methods to better understand the nature of lubricants and to better study which are the modifications occurring during their use. Simply transferring the well-known methodologies from mineral oils to vegetable oils would result in an unrealistic picture of these products. A preliminary chemical evaluation of base esters should detect defects that may cause problems during in-use operation. The composition of a material is a determining factor of its viscosity. When this composition is altered, either by changing the proportions of the component substances, or by the addition of other materials, a change in viscosity is quite likely.

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APPENDICES

APPENDIX 1

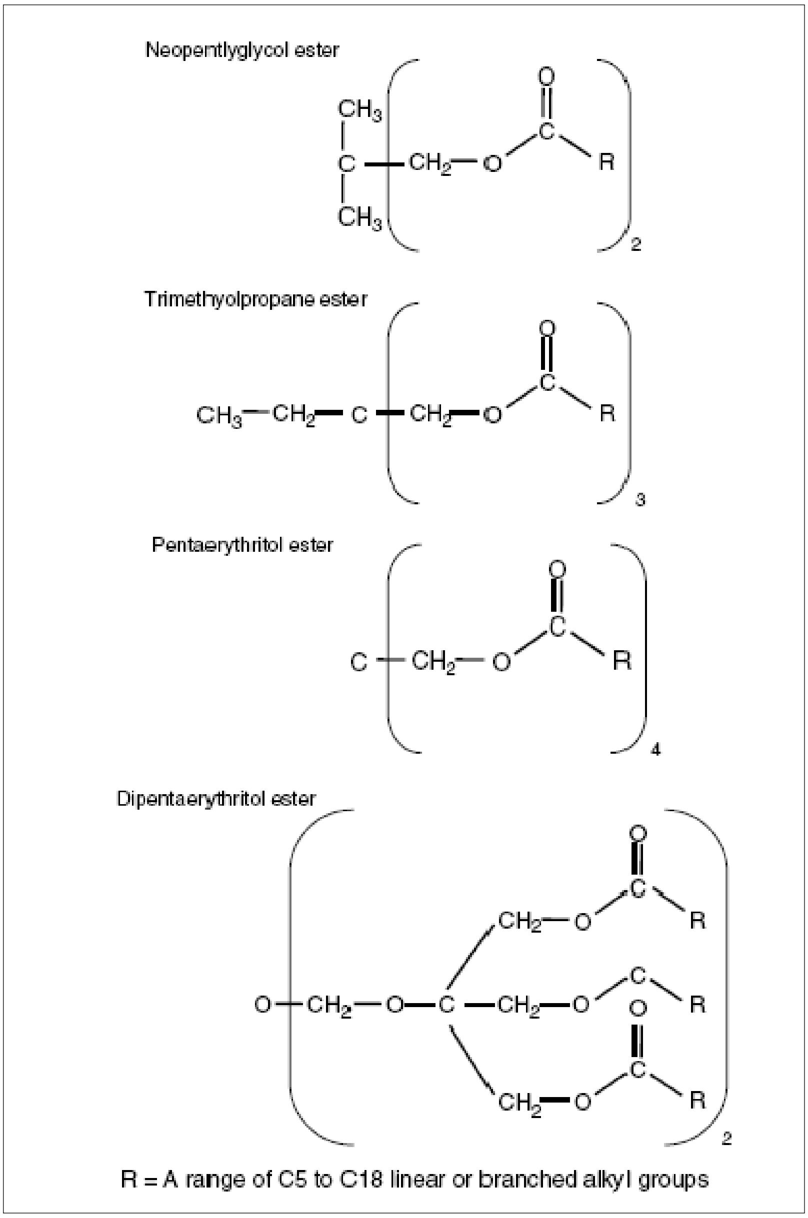
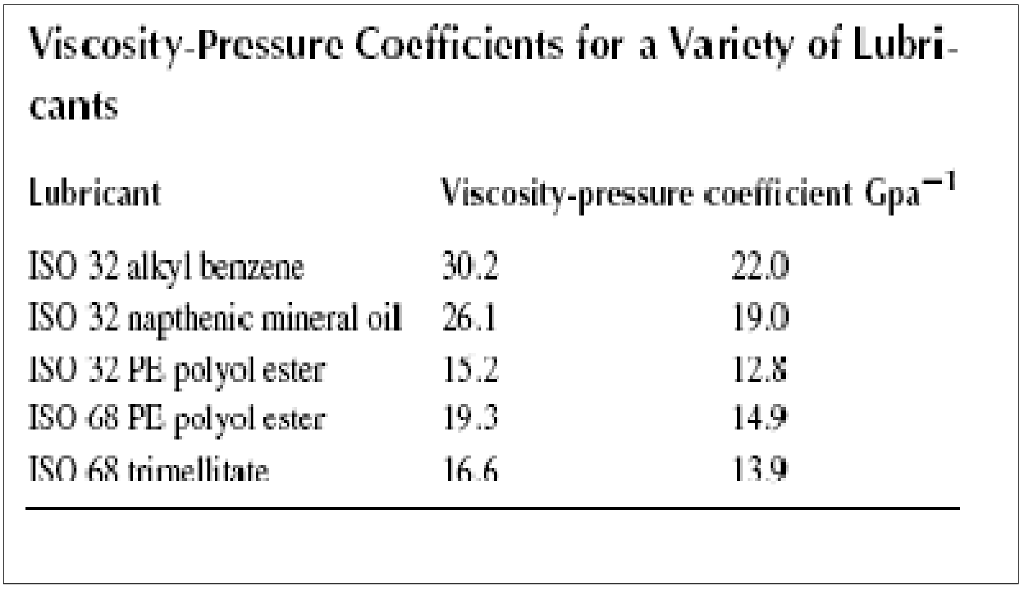


Figure A1: Chemical Structure of Polyol Ester

APPENDIX 2

Table A1: Viscosity-Pressure Coefficients for a Variety of Lubricants

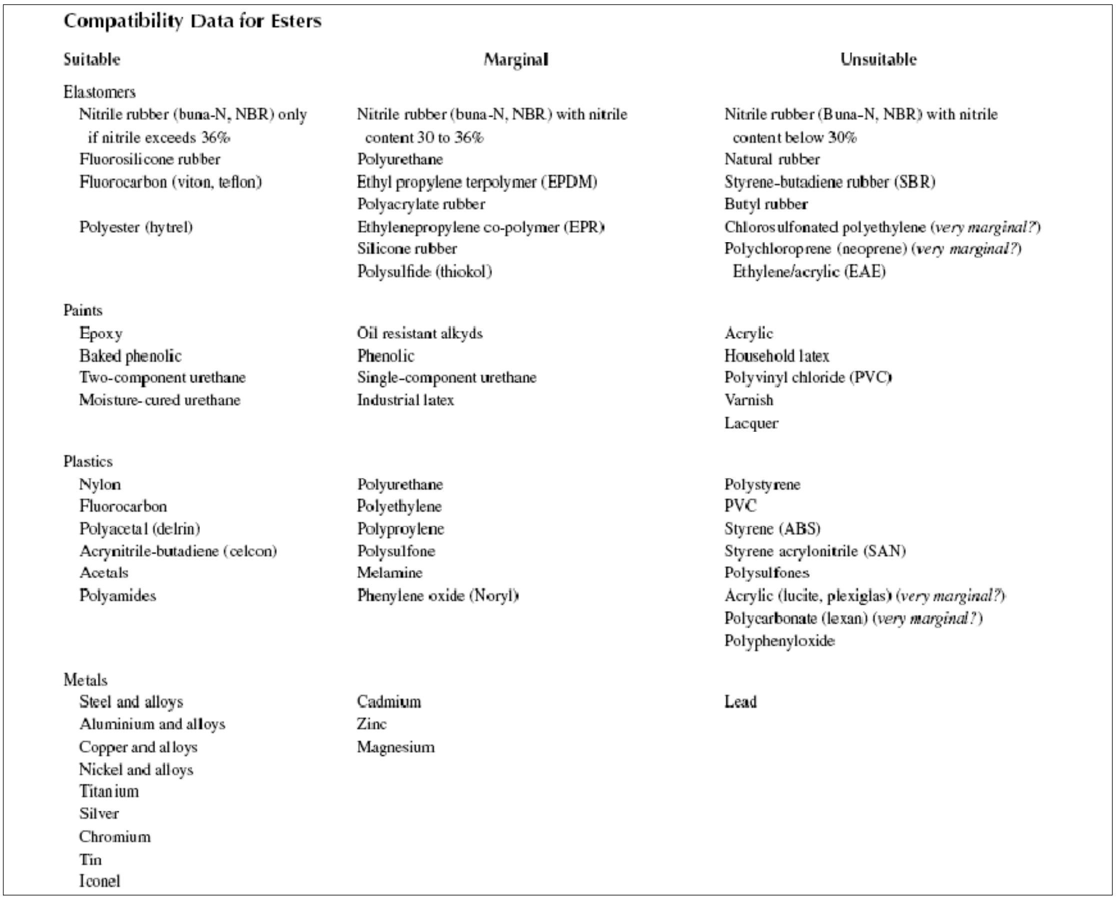


Viscosity-Pressure Coefficients for a Variety of Lubricants

Lubricant	Viscosity-pressure coefficient Gpa^{-1}	
ISO 32 alkyl benzene	30.2	22.0
ISO 32 napthenic mineral oil	26.1	19.0
ISO 32 PE polyol ester	15.2	12.8
ISO 68 PE polyol ester	19.3	14.9
ISO 68 trimellitate	16.6	13.9

APPENDIX 3

Table A3: Compatibility Data for Esters

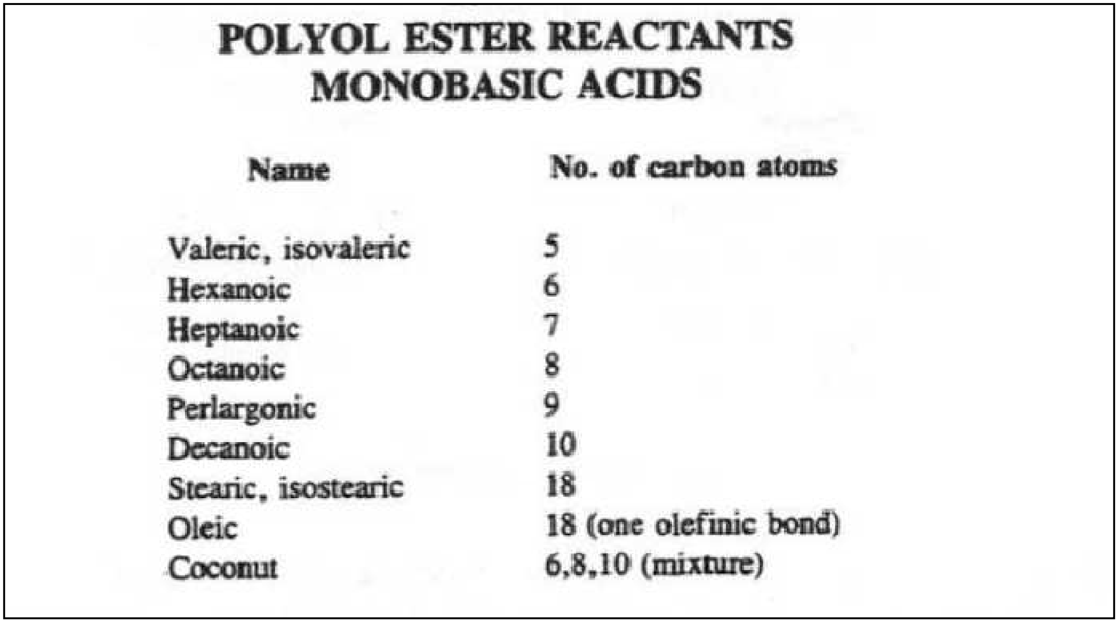


Compatibility Data for Esters

Suitable	Marginal	Unsuitable
Elastomers		
Nitrile rubber (buna-N, NBR) only if nitrile exceeds 36%	Nitrile rubber (buna-N, NBR) with nitrile content 30 to 36%	Nitrile rubber (Buna-N, NBR) with nitrile content below 30%
Fluorosilicone rubber	Polyurethane	Natural rubber
Fluorocarbon (viton, teflon)	Ethyl propylene terpolymer (EPDM)	Styrene-butadiene rubber (SBR)
	Polyacrylate rubber	Butyl rubber
Polyester (hytrel)	Ethylenepropylene co-polymer (EPR)	Chlorosulfonated polyethylene (*very marginal?*)
	Silicone rubber	Polychloroprene (neoprene) (*very marginal?*)
	Polysulfide (thiokol)	Ethylene/acrylic (EAE)
Paints		
Epoxy	Oil resistant alkyds	Acrylic
Baked phenolic	Phenolic	Household latex
Two-component urethane	Single-component urethane	Polyvinyl chloride (PVC)
Moisture-cured urethane	Industrial latex	Varnish
		Lacquer
Plastics		
Nylon	Polyurethane	Polystyrene
Fluorocarbon	Polyethylene	PVC
Polyacetal (delrin)	Polyproylene	Styrene (ABS)
Acrynitrile-butadiene (celcon)	Polysulfone	Styrene acrylonitrile (SAN)
Acetals	Melamine	Polysulfones
Polyamides	Phenylene oxide (Noryl)	Acrylic (lucite, plexiglas) (*very marginal?*)
		Polycarbonate (lexan) (*very marginal?*)
		Polyphenyloxide
Metals		
Steel and alloys	Cadmium	Lead
Aluminium and alloys	Zinc	
Copper and alloys	Magnesium	
Nickel and alloys		
Titanium		
Silver		
Chromium		
Tin		
Iconel		

APPENDIX 4

Table A4: Polyol Ester Reactants Monobasic Acids

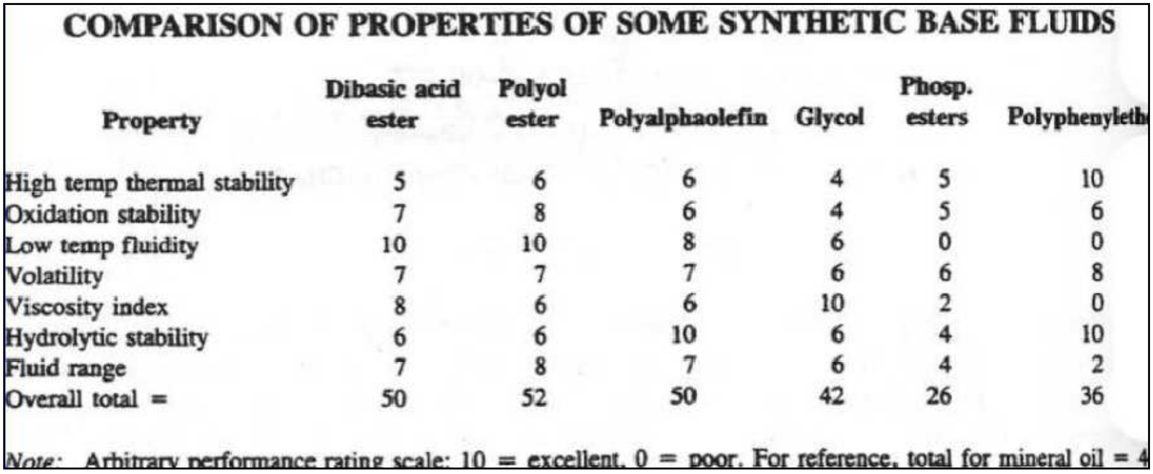


POLYOL ESTER REACTANTS
MONOBASIC ACIDS

Name	No. of carbon atoms
Valeric, isovaleric	5
Hexanoic	6
Heptanoic	7
Octanoic	8
Perlargonic	9
Decanoic	10
Stearic, isostearic	18
Oleic	18 (one olefinic bond)
Coconut	6,8,10 (mixture)

APPENDIX 5

Table A5: Comparison of Properties of Some Synthetic Base Fluids



COMPARISON OF PROPERTIES OF SOME SYNTHETIC BASE FLUIDS

Property	Dibasic acid ester	Polyol ester	Polyalphaolefin	Glycol	Phosp. esters	Polyphenyleth
High temp thermal stability	5	6	6	4	5	10
Oxidation stability	7	8	6	4	5	6
Low temp fluidity	10	10	8	6	0	0
Volatility	7	7	7	6	6	8
Viscosity index	8	6	6	10	2	0
Hydrolytic stability	6	6	10	6	4	10
Fluid range	7	8	7	6	4	2
Overall total =	50	52	50	42	26	36

Note: Arbitrary performance rating scale: 10 = excellent, 0 = poor. For reference, total for mineral oil = 4