



**UNIVERSITI TEKNIKAL MALAYSIA MELAKA**

**BIO-DEGRADABLE GREASE FORMULATION USING FRESH PALM OIL AS  
PRECURSOR**

This report is submitted in accordance with the requirement of Universiti Teknikal Malaysia Melaka (UTeM) for the Bachelor of Mechanical Engineering Technology (Maintenance Technology) with Honors

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## **APPROVAL**

This report is submitted to the Faculty of Engineering Technology of UTeM as a partial fulfillment of the requirements for the degree of Bachelor of Mechanical Engineering Technology (Maintenance Technology) with Honors. The member of the supervisory is as follow:

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## ABSTRAK

Produk yang berasaskan petroleum digunakan secara meluas dalam bidang industri. Walaubagaimanapun, petroleum yang sedia ada sekarang semakin berkurang. Ini akan membawa kepada peningkatan harga petroleum dan barang-barang yang berasaskannya. Untuk menghadapi masalah ini, kajian ini mencadangkan untuk mencipta jenis gris yang baru. Gris baharu ini akan menggunakan minyak kelapa sawit segar sebagai bahan mentahnya. Ini kerana minyak kelapa sawit adalah bahan yang boleh diperbaharui dan bio-terurai. Gris akan diformulasikan dengan menggunakan minyak kelapa sawit sebagai pelopor dan Lithium Hydroxystereate sebagai ejen pemekat. Zinc Dialkyldithiophosphates (ZDDP) juga digunakan sebagai tambahan untuk menambahbaikan ciri-ciri minyak dan dijadikan sebagai ejen anti-oksidan dan anti-kehausan didalam minyak asas. Ketiga-tiga bahan ini akan dicampurkan bersama dengan kepekatan yang berbeza untuk mencipta gris baharu. ZDDP akan digunakan sebanyak 2% berat dan nilai Lithium Hydroxystereate yang berbeza yang mengandungi sebanyak 5% berat, 10% berat, 15% berat dan 20% berat didalam setiap sampel. Formulasi gris yang baharu akan diuji pelincirannya dengan menggunakan 'Four-ball Tester' mengikut ASTM D2266 dan ASTM D2596 untuk menyiasat diameter parut kehausan dan tekanan melampau manakala "Scanning Electron Microscopy" (SEM) digunakan dalam proses pencirian untuk imbas tindak balas pada permukaan dengan resolusi yang tinggi. Kajian ini menunjukkan Sampel 2 dengan kepekatan 2% berat ZDDP dan 10% berat Lithium Hydroxystereate memperolehi nilai tribologi yang wajar berbanding dengan sampel yang lain. Ianya menunjukkan pekali geseran terendah pada 0.091 dan diameter parut kehausan terkecil pada 372 $\mu$ m manakala sampel 4 mempamerkan nilai beban yang digunakan tertinggi dalam ujian 'extreme pressure' iaitu 315Kg<sub>f</sub>. Pada pengakhiran kajian ini, gris bio-terurai yang baharu berjaya dicipta dengan mencampurkan 2% berat ZDDP dan Lithium Hydroxystereate ke dalam minyak kelapa sawit komersial.

## ABSTRACT

Petroleum based products were widely used as lubricant in the industry. However, to date it is found out that petroleum reserve is decreasing. This leads to the increase of petroleum and petroleum based products prices. To overcome this problem, this study proposes to develop a new type of grease. The new grease will use fresh palm oil as its raw ingredient. This is because palm oil is renewable and biodegradable. The grease will be formulated using pure palm oil as grease precursor and Lithium Hydroxystereate as thickening agent. Zinc Dialkydithiophosphates (ZDDP) also used as the additive to enhance the properties and also as anti-wear and anti-oxidant agent in the base oil. These three element will be blended together with different concentration to develop a new biodegradable grease. ZDDP will be used for each sample with concentration of 2wt% and different value of Lithium Hydroxystereate which contains of 5wt%, 10wt%, 15wt% and 20wt%. The formulated new bio lubricant will then be tested for its lubricity using a Four-ball Tester in accordance of ASTM D2266 and ASTM D2596 to investigate wear scar diameter and extreme pressure while Scanning electron Microscopy (SEM) used for characterization which to scan the behavior on the surface with the higher resolution. This study showed the Sample 2 with the concentration of 2wt% ZDDP and 10wt% of Lithium Hydroxystereate obtained the most desirable tribological among the prepared samples. It displayed the lowest coefficient of friction at 0.091 and the smallest wear scar diameter at 372 $\mu$ m while Sample 4 exhibited the highest applied load in extreme pressure testing which 315Kg<sub>f</sub>. In the end of this study, the new biodegradable grease was successfully developed by blending with 2wt% ZDDP and Lithium Hydroxystereate into commercialized cooking palm oil.

## **DEDICATION**

To my beloved family

To my respected supervisor, Qamar Fairuz Bin Zahmani

To my lecture, Muhamad Azwar Bin Azhari

To my group member, Muhammad Najib Bin Mohd Eza and Zuhayra Raihan Binti  
Zahisham

To my helpful friends.

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## LIST OF SYMBOL AND ABBREVIATIONS

%	-	Percent
ASTM	-	America Society for Testing and Materials
$\beta'$	-	Beta
C	-	Carbon
$^{\circ}\text{C}$	-	Degree Celsius
DBP	-	Dibutylphosphite
F	-	Force
$^{\circ}\text{F}$	-	
H	-	
HO	-	Hydroxide
HRC	-	Hardness C Rockwell
$\text{H}_2$	-	Hydrogen
Kg	-	Kilogram
$\text{Kgf}^{\text{A}}$	-	
L	-	Load
Li	-	Lithium
Mm	-	Millimeters
MoDTC	-	Molybdenum Dithiocarbamate
MoDTP	-	Molybdenum Dithiophosphate

N	-	Newton
O	-	Oxygen
PTFE	-	Polytetrafluoroethylene
Rpm	-	Rotation Per Minutes
S	-	Second
SEM	-	Scanning Electron Microscopy
Wt	-	
ZDDP	-	Zinc Dialkyldithiophosphate





# CHAPTER 1

## INTRODUCTION

Grease is substance that used to remain in contact or separate two or more moving surfaces from each other's direct contact. The word of grease can also be derived as fat in Latin Crassus, and the first grease was made from animal fats and pitch. For example, a combination of lime (calcium carbonate) and olive oil was used by ancient Egyptians and Romans as a lubricant in plain carriage bearings. In the 1859, USA have invented the grease which was use the mineral as based oil (Lugt et al., 2013). Meijer et al (1996) stated the one that got into the commercial product in 1992 which is the newest evolution grease product in 1992 is the polymer grease.

The grease is the lubricants which are commonly use in rolling element bearing application (Kanazawa et al, 2017). Grease is classified as the semi-solid lubricant to minimize the friction between the contacted surface (Barriga, 2006, Udonne, 2011 and Akpan et al, 2011). The advantage of the grease is to remain the space between the two or more surface to prevent the component deterioration. Greases are created by distribute the thickening agent into the base oil in the certain blend temperature. Thickener is the mechanism in the grease to remain oil in the semi solid state. Lugt et al (2013) stated the thickener is the mechanism to ensure the grease to maintain in semi solid state by keep the base oil in its fiber structure. In addition, this allow the grease have the upper hand in the usage of grease in the machinery such as sealing agent and the lubricant retention abilities. Gow (1997) stated the grease can act as seal and stay remain in one place due its semi-solid condition. This behavior will prevent the any dirt or impurity from entering the system. Besides that, the thickener also provides the consistency to the grease which to avoid the grease leaking occur from the bearing. This purpose also make grease easier to use than fluid lubricant and this is the reason the usage of the grease as the lubricant

have been applied about 80-90% at the rolling element such as bearings, (Lugt et al., 2013). Grease also can minimize the shock and the noise produced from the machine instead act as seal and prevention from corrosion (Abdulbari et al, 2011).

In the ancient time, the grease is known as the oldest forms in the lubrication world. In that day, the greases were made by the lime, mutton fat and beef fat which they are all environment friendly and biodegradable materials (Khodijah et al., 2012). However, the mineral start to take over as the main source of based oil of lubricant as the increasing friction in the industrial machine and the problems on automobile (Awoyale et al, 2016). The emergence of petroleum or mineral based lubricants at 19<sup>th</sup> century, also known as Industrial Revolution (Suhane, 2012) widely used at every industrial machines as the transformation of technology in order to improve the economic and culture condition of the time. Mineral based lubricants provide the massive job creation and increase the profit of the industries as well because the process to find the mineral may be expensive. By any means, the emergence of mineral based product causes the environment pollution due this unrenewable source is non-biodegradable. However, bio-based material as the main lubricant are indispensable due the increasing of industrial and automotive lubricant in recent years (Awoyale et al., 2016). Drake (1991) state the vegetable oil become popular due have the speciality of non-toxic and biodegradable material that have the potential to replace the mineral based oil as the main lubricant in grease making.

## **1.1 Composition of Grease**

According to (Sukirno et al, 2009), grease is composed through the addition of three elements which is a base oil, a thickening agent and an additive. The following subsection will elaborate each element.

### 1.1.1 Base Oil

The use of the mineral as the parent base oil of lubricant has been used widely in the industry. However, the usage of mineral based lubricant will contaminate the environment which effect the greenhouse and he warming global issue have been increasing lately due this product is non-degradable and toxic (Castro et al, 2006,and Zulkifli et al., 2013). The vegetable oil become popular that potential to replace the mineral based oil as the lubricant since they are biodegradable and environment friendly.

Biodegradable grease is the lubricant created from the renewable source which using the vegetable oil as the based oil of lubricant that will not harm to the environment. According to (Azhari et al, 2014). There are two types of vegetable oil which are edible and non-edible. Edible is the oil type can be eaten as a food or processing food such as palm, corn, sunflower, olive, peanut etc. Non-edible vegetable oil such as mahua, rice bran, karanja, Jatropha and linseed oils (Sevim et al, 2006). There are several advantages of using the vegetable oil as the based oil lubricant which are they are non-toxic, biodegradability, renewability of resource, low cost of application and high viscosity index (Reddy et al, 2014). There are many researchers choose the vegetable oil as the base stocks of lubricants due their excellence characteristic such as has the higher flash and fire point, high thermal stability and better biodegradability than the mineral based lubricant (Azhari et al, 2016a). However, Liu et al (2014) stated the vegetable oil is lacking in the oxidation stability and will be the problem in order to develop the vegetable oil. This problem will lead to increase the oil acidity, viscosity value, corrosion and volatility.

### **1.1.2 Additives**

(Azhari et al, 2015) mentioned high unsaturated fat acid in the vegetable oil is the factor of oxidation problem where it is less collaborative with the produced oil in order to stabilize the oxidation. In order to overcome the problem in stability of oxidation, the researchers have found the best solution which they use antioxidant additive Zinc Dialkyldithiophosphate (ZDDP) (Azhari et al., 2016a). According to (Erhan et al (2006), the ZDDP have magnificent properties as the antioxidant agent.

With the additive of lithium hydroxystereate as the thickening agent, this will increase the viscosity of the lubricant and remain the lubricant in semi-solid lubricant. Thickener is the most important component in developing the grease. The typically thickener consist of three which are calcium, sodium and lithium hydroxystreareate. However, the lithium hydroxystereate most widely used as the thickening agent in the grease. Kudryavtsev, (2016) stated the lithium hydroxystereate reveals the higher in the oxidation stability close to about 200°C and also have the water resistance. The lithium also have speciality at the high and low temperature. For example, the lithium based grease have higher melting point which is more better in the high temperature than the calcium based grease approximately of 190 to 220°C (Kudryavtsev, 2016).

## **1.2 Problem Statement**

Mineral based products were widely used as lubricant in the industry a long time ago. However, the mineral based lubricant is created from the petroleum which is non-renewable and non-biodegradable lubricant. Furthermore, to date it is found out the mineral reserve is decreasing and the demand of petroleum from industries are incredibly increase until now. Therefore, the price of the petroleum had increased every each years until now.

The use of petroleum as the lubricants has caused the environment pollutant become the major concern lately. According to Azhari et al., (2015), the issues of environmental that related to non-biodegradable and petrochemical began to increase due the waste oil is very hard to dispose. This problem occur when the wasted oil and the following disposal will acute the environment pollutions. Miller et al., 2007 mentioned the combustion of mineral oil as lubricant will give the side effect which discharge the metal traces such as the calcium, magnesium, iron particles and zinc. These problems will lead the environmental degradation. The common pollutions are in hydraulic, agriculture, mining and the petrochemical industries (Mahipal et al, 2014). Sapawe et al (2014) stated the petrochemical which in the mineral based lubricant have the higher toxic and flammable which can harm the surrounding hazardly. Besides that, aquatic life and land ecosystem will threatened if the disposal of the non-degradable mineral oil still continued (Ssempebwa and Carpenter, 2009). The researchers start to look forward of the vegetable oil as the renewable source and environment friendly that potentially to replace the the limited or non-renewable crude petroleum and the inimical the environment (Zulkifli et al., 2013; Asadauskas and Erhan, 1999; Beran, 2008). Development of vegetable oil as the bio-degradable lubricant and renewable source which have the excellent characteristic become the most suitable substitute to mineral oil as lubricant.

The vegetable oil which with more eco-friendly resource will be the best solution to replace the mineral oil as the main based lubricant in the industries. According to (Anand et al, 2014), vegetable oil commonly have the desired properties such as high in viscosity index, excellent lubricity, high flash point, bio-degradability and lower toxicity have been consider to use as the base lubricant oil. The main problem of using the vegetable oil as the lubricant is the oxidation stability very poor and will cause the increase of oil acidity, viscosity value, corrosion and volatility.

However, many researchers have come up with the best solution to resolve the problem of oxidation stability which use the additive Zinc Dialkyldithiophosphate (ZDDP) and lithium hydroxystereate as addition into the vegetable oil to develop grease.

According to Erhan et al, (2006), the ZDDP have the best performance as the antioxidant agent. ZDDP also have the good performance in reducing the wear and friction. (Farhanah and Syahrullail, 2015) mentioned the ZDDP acts as the anti-wear and antioxidant agents. Litium hydroxystereate also known as the lithium soap will use as the thickening agent for increasing the viscosity of vegetable oil. Besides that, lithium soap also have the speciality which is can apply in high and low temperature and will help the in development of bio-grease with the speciality in the high and low temperature condition. The combination of vegetable oil and ZDDP and Lithium Hydroxystereate started become popular among the researchers and it is believed that this new bio-lubricant can surpass the mineral based grease in reducing the friction and wear.

Therefore, this study proposes to develop a bio-lubricant with the right amount concentration of ZDDP as the additive agent into the vegetable oil to get the best behavior of wear and COF in order to surpass the mineral based lubricant.

### **1.3 Objectives**

Based on the problem statement, the objectives of this study are listed as follows:

- i. To develop a new biodegradable grease using fresh cooking palm oil.
- ii. To test the developed new biodegradable grease for its lubricity performance

#### **Scope**

- i. Developing new biodegradable grease using fresh palm oil, ZDDP, Lithium Hydroxystereate.
- ii. Testing the lubricity performance of biodegradable grease using four ball tester
- iii. Characterizing of newly developed biodegradable grease using SEM.

## **CHAPTER 2**

### **LITERATURE REVIEW**

#### **2.1 LUBRICANT**

Lubricant is used to reduce friction and wear in order to extend the life of the equipment. Lubricant also reduces heat when two or more surfaces sliding on each other in relative motion. Ahmed and Nassar (2013) mentioned, lubricant is also used to reduces oxidation and prevent the equipment from rusting. Therefore, lubricant also made into the insulation in transformer application and transfer the mechanical power in hydraulic fluid power application and as a seal to prevent from foreign particles enter the system. Petroleum base products have been found as lubricant for almost every situation due they excellent in metal wetting and have viscosity characteristic that have a substantial film requires. Besides that, the petroleum base stocks have ability of water resistance, preventing from rusting, natural adhesiveness, have excellent stability of thermal and able to transfer the heat caused by friction away from lubricated parts (Ahmed and Nassar, 2013). However, the petroleum is the created from the non-renewable source which is this source will be finished sometimes. Furthermore, the mineral source is began to decreasing and the demand of petroleum from the industries are increasing until now. This will caused the price of the petroleum increased every each years until now. However, the emergence of vegetable oil became a discussion among the researchers which it is a renewable source and have the potential to replace the mineral based products.