



**UNIVERSITI TEKNIKAL MALAYSIA MELAKA**

**FORMULATION OF ANTI-FRICTION BIODEGRADABLE  
GREASE USING PALM OIL WITH ADDITION OF LITHIUM  
HYDROXYSTEARATE AND ZINC DIAMYL  
DITHIOCARBAMATE (ZDDC)**

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

by

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**BORANG PENGESAHAN STATUS LAPORAN PROJEK SARJANA MUDA**

**TAJUK: Formulation of Anti-Friction Biodegradable Grease Using Palm Oil with Addition of Lithium Hydroxystearate and Zinc Diamyl Dithiocarbamate (ZDDC)**

**SESI PENGAJIAN: 2019/20 Semester 1**

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## **APPROVAL**

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

.....  
(Qamar Fairuz Bin Zahmani)

## ABSTRAK

Minyak berasaskan petroelum adalah tidak terbiodegradasi dan oleh kerana prosedur pelupusan yang kompleks menjadikannya tidak mesra alam. Oleh itu, kajian ini akan menumpukan kepada pembangunan bio-pelincir gris baru sebagai alternatif kepada minyak pelincir berasaskan minyak mineral di dalam pelincir gris. Dalam kajian ini, empat jenis sampel telah disediakan dengan berbeza tahap kepekatan pemekat. Bio-pelincir gris ini akan dihasilkan dengan menggunakan minyak masak komersial kelapa sawit dan ditambah ZDDC sebagai aditif dan Lithium Hydroxystearate sebagai pemekat. Sampel akan diuji dan dicirikan dengan menggunakan ujian yang akan mengikut piawaian ASTM untuk menentukan sifat pekali geseran diameter parut terhakis, dan tekanan yang melampau dengan menggunakan ujian empat bola sementara diameter parut terhakis akan diimbas dibawah SEM. Hasil dari ujian dan pencirian bio-pelincir gris yang baru dibangunkan menunjukkan minyak kelapa sawit dengan 2 wt% ZDDC dan 10wt% Lithium Hydroxystearate memberikan keputusan yang diinginkan daripada sampel yang dimana ia memberi nilai paling rendah pekali geseran pada 0.090 dan diameter parut terhakis 765.3  $\mu\text{m}$ . Nilai paling tinggi untuk ujian tekanan yang melampau terlihat pada 315 kgf. Kesimpulannya, prestasi yang lebih baik daripada bio-pelincir gris yang baru dibangunkan ini akan mendorong penghasilan pelincir alternatif yang boleh digunakan untuk industri hari ini seterusnya menukarkan penggunaan minyak pelincir berasaskan mineral.

## ABSTRACT

Petroleum based lubricant is a non biodegradable and due to its complex disposal making it non-environmental friendly. Thus, this study will be highlighted on developing a new bio lubricating grease as an alternative to substitute the mineral based oil in the lubricating grease. In this study, four samples of lubricating grease were prepared with different concentration thickener. The bio-lubricating grease were developed by using a fresh commercialized cooking palm oil with addition of ZDDC as additive and Lithium Hydroxystearate as thickener. The samples were tested and characterized in accordance to ASTM test methods to determine the coefficient of friction, wear scar diameter, and extreme pressure. The COF and EP were characterized using four ball test method while WSD was observed under SEM. The result from test and characterization of the newly developed bio-lubricating grease showed palm with 2 wt% of ZDDP and 10wt% of Lithium Hydroxystearate exhibit the desirable result from the other samples which give the lowest coefficient of friction at 0.090 and the wear scar diameter of 765.3  $\mu\text{m}$ . The highest load resist under extreme pressure test also was observed at 315 kgf. As a conclusion, the positive performance of this newly developed bio-lubricating grease resulted in producing an alternative lubricant which can be suggested to be applied to industry today as substitution of using mineral based oil lubricant.

## **DEDICATION**

I would like to dedicate my thesis to my beloved parents and my siblings.

## **ACKNOWLEDGEMENT**

I would like to give my full gratitude to Allah S.W.T because with his grace and kindness I can carry out this study. Apart of that, I would like to dedicate my thanks to my supervisor, Mr. Qamar Fairuz Bin Zahmani for giving me the opportunity to do this study under his guidance. Apart from that, I would like to thank my parents and siblings for the endless support throughout this journey. My thanks also to my teammates that have always helping me in any condition. Lastly, thanks to anyone that help me directly and indirectly during this study. I believe this thesis would not have been possibly done without all of these support and help.



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## LIST OF ABBREVIATIONS, SYMBOLS AND NOMENCLATURE

ASTM	-	American Society for Testing and Materials
BC	-	Before Christ
CO	-	Carbon monoxide
MCT	-	Medium chain triglycerides
MoDTC	-	Molybdenum dithiocarbamate
MoDTP	-	Molybdenum dithiophosphate
MoS <sub>2</sub>	-	Molybdenum disulphide
PAO	-	Polyalphaolefin
PIB	-	Polyisobutylene
PIO	-	Polyinternalolefin
PKO	-	Palm kernel oil
PCTFE	-	Polychlorotrifluoroethene
PTFE	-	Polytetrafluoroethylene
PV	-	Present value
PVF	-	Polyvinyl fluoride
SAE	-	Society of automotive engineer
SEM	-	Scanning electron microscopy
SI	-	International system of unit (Systeme internationale)
SiO <sub>2</sub>	-	Silicon dioxide
VI	-	Viscosity index
ZDDC	-	Zinc Dialkyldithiocarbamate
ZDDP	-	Zinc Dialkyldithiophosphate

# CHAPTER 1

## INTRODUCTION

### 1.1 Introduction to Lubricating Grease

Lubrication has been vital since the early time of human beings becoming expertise in transportation and machinery application. Since more development in improving the both applications, a lubricant was needed to reduce the effort. Most people did not acknowledge with the principles of how lubricant works. Lubricant is known as a substance that is used to reduce friction when two surfaces are contacted in motion. Friction needs to be reduced as it contributes to the generation of heat as well as wear. Bannister (1996) stated that by forming a fluid film between the surfaces, lubricant helps to reduce friction which eventually reduce the generation of heat and wear. A study done by Anderson (2016) stated that lubricants was used by humans since the 19<sup>th</sup> century where the rise of petroleum industry makes it to be used dominantly. Petroleum itself was widely used since the ancient time. Peoples in ancient time used petroleum as source of lighting and embalming. While the American Indians used petroleum as a remedy in medical purpose. Proportional to the development of technology and demands, mineral oil started to be used widely as it gives more benefits in many terms (Anderson, 2016).

Lubricating grease has been a need since the ancient times as the Egyptians used animals' fat to reduce friction. Lugt et al, (2013) stated that the word grease itself is originated from a Latin crassus which means fat. According to Duncan (1998), lubricating grease has witnessed a long continuous change from a raw animal fat, vegetables oils and water to the modern lubricating grease. Lubricating grease cannot be more than a thickened lubricant. Thousand years ago calcium-based soap was , the first soap used to thicken grease and it succeeded in ruling the market the market for almost half of a century (Honary & Richter, 2011a). During the eras of Egyptian and

Roman they had created a calcium-based grease which is from the combination of lime or calcium carbonate and olive oil to be used in plain bearings. However, good lubricating greases were not available until the development of petroleum-based oils in the late 1800's. In 1859, The Americans discovered mineral oil to be used in most lubricants with addition of calcium soaps but it only last for a short term until new greases were developed based on lithium (Lugt et al, 2013).

Lubricating grease can be defined as a solid or semi-fluid lubricant that consists agent of thickening and other ingredients that can enhance special properties in a liquid lubricant. According to grease is a lubricant which has been formulated with combination of metallic soap in order to maintain the contacts between the moving surfaces and able to resist from leakage or be squeezed out when under gravity or pressure. Depending on the application, grease can provide several advantages in comparing with other fluid lubrication. Greases provide a physical seal which protects it from contamination, high resistance from water, and can stay in place for an application in any position (Honary & Richter, 2011a). Grease is suitable in any applications due to its mechanism, type of sealing as it contributes a big help in the prevention of lubricant loss or contamination ingress (Abdulbari et al. 2011).

Greases are produced by combining three major components which is base oil, additives and thickener. Base oils comprise the largest components in creating grease. According to Kim et.al, (2009), most grease comprise of 80-90% base oil. The choice of base oil in creating grease may be mineral oil, synthetic oil, or any fluid that offers the lubricating properties. All base oils have their own characteristics that offers different performance in lubrication. Mineral oil is a derivation of crude oil that is produced from the oil's refining process. Synthetic oil is an oil that is produced through synthesizing process with various formulation that may not be achievable in a mineral oil, while vegetable base oil is a derivation of plant product and have been chosen primarily for renewable and environmental interests(Honary & Richter, 2011b).

Additives plays several roles in lubricating grease which includes contamination control. Additives acts as a seal that helps to prevent the metal surface from in contact with water(Wang et.al, 2017). This will prevent the production of rust

and preventing damage to the metal machine surfaces. Besides, it could enhance the existing properties of the lubricating grease additives. In choosing the suitable additives to be added in the lubricating grease, the applications requirements need to be determined. For example, the speed of the metal parts needing lubrication, or the mechanical loads applied on the parts. All of these factors can determine the required viscosity (Papay & Petroleum, 1998). The most common additives are antioxidants, anti-corrosive agents, anti-foaming agents, anti-wear agents and additions for use at very high pressure (EP). Oils will be oxidised when are in used at high temperature and in contact with the air. Substances will be formed which can changed the viscosity of the oil and led to corrosion. Antioxidants indirectly improve the stability of the besides preventing the oils from oxidizing (Syahrullail et al., 2013).

Thickeners are one of the main components required in preparing greases. Greases are classified based on their thickener type as it forms about 10 – 15% of the grease (Lugt et al, 2013). The material of the thickener controls the consistency of the grease. The thickener can be any material that used to combine with the base oil as it will be the reason to produce the solid to semi-fluid structure. It is often referred to as a sponge that holds the mixture of base oil and additives. The common type of thickening agent used in current grease is lithium based. Lithium compound is being selected because of their outstanding criteria which is high dropping points and excellent load-carrying abilities. The most common component used in grease are mixtures of mineral oils and lithium soaps. According to (Earle, 2009), Lithium based lubricating grease was first introduced in 1940s and was a huge debut for lubrication industry. The grease was formulated from combination of lithium compound and hydroxystearic acid which exhibits superior properties compared to another chemical compound. Kudryavtsev (2016) stated that when lithium hydroxide is heated with fat, a soap containing lithium (lithium stearate) will be formed as it has the ability to thicken oil and also increasing the viscosity of oils. Besides, its properties which is thermal stability and water tolerance make it as a renown lubricating grease. Kudryavtsev (2016) also stated that greases based on lithium soap are recommended due to their good characteristics such as resistance to water, perform well at both high and low temperature, as well as the mechanical and oxidative stability. The lithium 12-hydroxystearate are widely used for preparation of grease as it can exhibits high



stability to oxidation approximately to about 200°C. There is other grease like complex-soaps, non-soap-based grease but still cannot defeat the lithium thickener (Honary & Richter, 2011a).

## 1.2 Problem Statement

As stated in earlier section, current industrial uses mineral based products in machinery application and pollution and environmental ill's issues has arisen recently. Mineral oil can be harmful due to its toxicity and exposure to environmental that might lead to pollution of both aquatic and human ecosystem (Azwar, Faisal, Rashid, & Rahimi, 2015). Most of current mineral oil is a derivation from petroleum and not adaptable to the environment due to its non-biodegradability characteristic (Shahabudin et al., 2013). Plus, petroleum is a non-renewable resource that can run out in the future. This issue has drawn the concern of lubrication industry to find alternatives by developing lubricating grease based on renewable sources. The use of biodegradable and renewable sources can help to maintain the environment and reduce the demand on mineral sources in the future (Azhari et al., 2015). Mineral oils are petroleum that consists of hydrocarbon derived by distillation of crude oil. These are usually a product obtained by the refining process and extracting of gasoline and other petroleum products. The extraction process is very high in cost and are not conventionally done. Besides, global mineral oil or crude oil reserves are depleting rapidly. Hence, it takes millions of years to reproduce and resulting to increment of price in crude oil (Afuape & Hughes, 2015)

Vegetables oil is said to be the perfect candidates as an alternative to mineral oil for lubricants formulations. as the conventional lubricating oil today can be replaced with vegetables oils (Chauhan and Chhiber, 2013). The oil is chosen because they are renewable, cost effective and eco-friendly (Azhari & Harun, 2016). However, there are some disadvantages of using vegetables oils as lubricant. According to a study found by (Liu et al., 2014) one of the limitations of using vegetables oils is they have poor oxidation stability, low temperature viscosities, and hydrolytic instability problems associated with the triglyceride. This problem can be said as critical because it causes an increase in oil acidity, viscosity, corrosion and volatility that can challenge the

capability of its property as lubricant. Vegetable oil have poor stability of oxidation because the level of unsaturated fatty acids in vegetable oil is very high in content (Azhari et al, 2014). The oil will oxides quickly while in use if not treated properly. Chemical modification of vegetables oils with addition of antioxidant agent can overcome this problem(Azhari, Fathe'li, Aziz, Nadzri, & Yusuf, 2015). However, this may be increasing the cost of developing the new lubricating grease. Another limitation faced by using vegetables oils is their high pour point. According to Azhari et al. (2015) vegetables oils comprise higher flash point as compared to mineral oil. Mineral oils responded better to pour point depressant additives as these were developed for the paraffin waxes found in mineral oils, not in most vegetable oils. The limitations can be overcome with additional of additives in the lubricating grease. As mineral oils also cannot meet the needs of most lubrication performance without additives Gawrilow (2004).

In order to overcome these problems, an additive is selected to be blended with the vegetable oil. A few studies found by few researchers stated/informed in their studies/ that an antioxidant additive Zinc Diamyl Dithiocarbamate (ZDDC) can be a solution regard to vegetable oil as lubricant. This is because ZDDC has an outstanding performance as antioxidant agent (Erhan et al. 2006). Apart from that, there is another additive being added into the lubricant oil to enhance the properties of the oil. Lithium Hydroxystearate is added into the lubricant oil as thickening agent and mineral base oil stocks. A few samples test will be prepared and carried out to test the effectiveness of both additives in the vegetables oil. The results then will be compared with current mineral oil.

### **1.3 Objectives of Research**

From the problem statement stated above, the objectives of this study are stated below:

1. To develop new biodegradable grease with addition of thickening agent and anti-friction agent.
2. To test and characterize the newly developed biodegradable grease.

### **1.4 Scope of Research**

In order to achieve the objectives, some scopes for this study have been constructed below:

1. Preparing biodegradable grease by using fresh cooking oil (Palm Oil) with addition of Lithium Hydroxystearate as thickening agent and Zinc Diamyl Dithiocarbamate (ZDDC) as antioxidant agent.
2. Testing the newly developed biodegradable grease by conducting four ball tests.
3. Characterizing of newly developed biodegradable grease using SEM.

## **CHAPTER 2**

### **LITERATURE REVIEW**

#### **2.1 Lubricant**

Basically, lubricant is a material in form of grease or oil that are used in most industrial field to ensure when two surfaces are not in contact to each other when in relative motion. Lubricant can work as a friction, heat and wear reducer by forming a fluid film in between the surfaces. Lubricant also used to lessen the oxidation and help to protect the machine components from rust. The main purpose of lubrication is to relief the wear and heat when two surfaces are contacted in relative motion (Ahmed and Nassar, 2013). Though both phenomena are permanent and cannot be eliminated, but they can be minimised to an acceptable level desired in corresponding application. Lubrication is meant to prevent damage of friction surfaces such as scuffing, together with scoring, seizure (Bart et al, 2012b). Anderson, (1991) reported in their studies that back then in 1400 B.C., lubricant is produced from the combination of calcium and fats producing lubricating grease. Though wear and heat cannot be demolished permanently, but it can be reduced to an acceptable condition by improving their properties. This is because both heat and wear phenomenon are influenced by friction. Hence, both effects can be overcome by reducing the coefficient of friction between the two surfaces (Ahmed and Nassar, 2013).

Modern lubrication was found by a researcher Osborne Reynolds in 1842-1912. He first discovered when lubricant is applied to a rotating shaft with bearings and cases, the shaft pulled a lubricant wedge which make the two surfaces separated constantly as the shaft gained velocity. This is because lubricant is viscous producing a liquid pressure in the wedge that is adequate to keep the surfaces separated in ideal conditions (Straffellini, 2015). In 1902 Stribeck come out with a theory which categorizes the friction in three different lubrication regimes (boundary, mixed, and

hydrodynamic). The regimes show as a function of the operating parameters, which includes normal applied load  $P$  to the contact, sliding speed  $N$  and viscosity  $\eta$  of the lubricant. The Hersey number (viscosity  $\times$  sliding velocity/applied pressure) is dependence to the lubricant film thickness between two matting surfaces (Bart et al., 2012b). Figure 1 summarizes the lubrication regimes by describing the relationship between speed, load, viscosity of oil, film thickness and friction which is known as Stribeck curve.

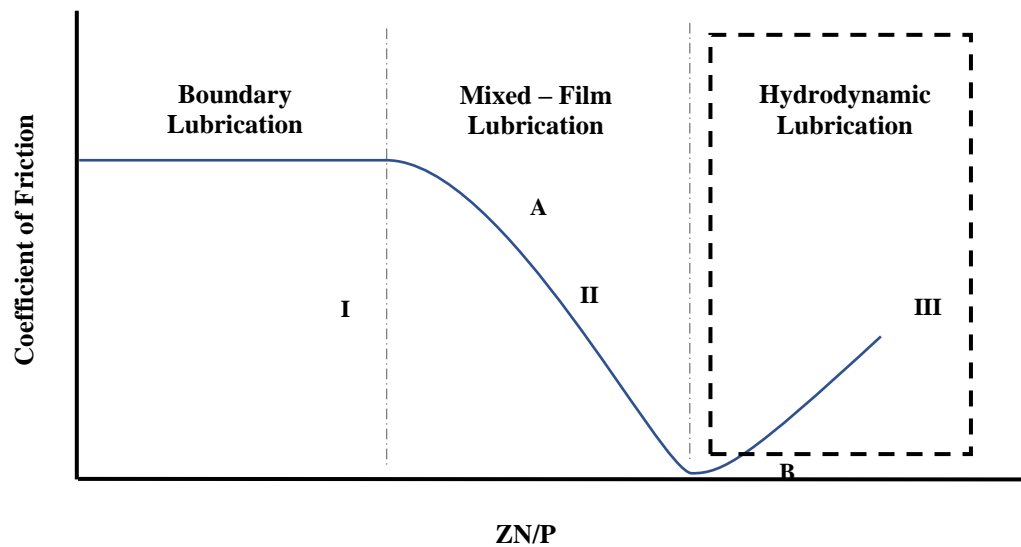


Figure 2.1 Stribeck Curve

The regimes are separated to three different zones by points A and B. At point A the oil film thickness reduces virtually to zero. While at point B the thickness of the oil film is greater enough to ensure that the surface between the shaft and bearing are not contacted (Straffelini, 2015).

### 2.1.1 Boundary Lubrication

Boundary lubrication regime in other word is a lubrication under liquid conditions where two solid surfaces are so close together which possible for the opposing asperities to contact to the surface. It occurs when there is a lacking fluid viscosity to prevent the surface contact. The layers of the film are very thin which means there is no contribution of hydrodynamic during this condition. If the layer is too thick, it is impossible for the lubricant films to be separated between the surfaces (Bart et al., 2012b). One of the characteristics of boundary lubrication is the layers are created from surface - active substances and chemical reactions. Boundary lubrication is very efficient when there is no presence of fluid film between the rubbing surfaces, the thickness of the film is relieved temporarily to replace the contact between wear surface high points or asperities (Straffelini, 2015). Unlike other regimes, friction and wear under boundary lubrication regimes are very difficult to handle as it can shortened the life of components (Bart et al., 2012b). The friction and wear in boundary lubrication are identified by interaction between the solids and the liquid (Straffelini, 2015).

The occurrence of boundary lubrication regimes often found in mechanical equipment application under high-load and low speed conditions. According to Bart et al., (2012) the term lubricity reflects to the slipperiness of lubricant films formed in boundary lubrication. In determining the friction of the surface, a few factors is taken into consideration such as properties of the contact surfaces and the lubricant been applied. Some researchers have investigated that role of hydrocarbons on boundary regimes did absorbed solid surfaces. Besides, the theories of boundary films have been applied in formulation of lubricants, greases and additives for lubricants. Straffelini, (2015) in his study stated that greases are the most common boundary lubricant because greases have the properties that are desired in boundary lubrication. Liquid lubricant is not really desirable in boundary lubrication, but solids and semi solids lubricants are very desirable boundary lubricants. This is because the type of lubricants mentioned earlier have great properties which is strong

chain attraction, low shear resistance and high temperature tolerance. A good boundary lubricant must be able to maintain the strong attachment even under high temperatures and extreme pressure conditions (Bart et al., 2012b).

Bart et al., (2012) stated that vegetables oils are very efficient in boundary lubrication as it contains high polarity which gives strong interactions between the lubricated surface. The level of performance for boundary lubrication is influenced by the attraction between the molecules in the lubricating oil and its possible reaction on boundary lubrication performance. Though the oil has an issue with the level of unsaturated fatty acids which might affect the performance of the boundary lubrication, however this matter can be countered by uprising the level of the concentration of oxygen in the oil to enhance the boundary performance (Azhari et al., 2015). Moreover, the boundary lubricants performance is dependence to the molecular structure of the base oil and additives. According to Bart et al., (2012) lubricants that operates under boundary conditions must have the ability to possessed the lowest coefficient of friction between the oil and matting surfaces. Hence, the property of the oil can be enhanced with the additional of anti-wear agents in the formulation. This is because most of lubricating oils are often used in heavier machine applications where boundary lubrication is very necessary. Therefore, the tribological performance of lubricating oil is closely related to the capability of film formation and the interaction strength with rubbing surfaces at boundary lubrication(Woydt & Wäsche, 2010).

## **2.2 Classification of lubricant**

Lubricants can be grouped in several ways which is solid (graphite, molybdenum disulphide etc.), semi-solid (grease), and liquid. Most of the types of lubricant mentioned earlier have their own characteristics in terms of its viscosities and other physicochemical characteristics.

### 2.2.1 Solid Lubricant

Solid-shape lubricant usually from a material in a form of powder or thin film that reduces the friction and wear of two contact surfaces and helps protect against damage. (Bart et al., 2012a) The application of solid materials based lubricants is dependence to the nature of the two surfaces. Solid lubricant applications are common seen in real life condition such as things on the road surface can be a lubrication to the vehicles as it work to reduce the friction between tyres and road surface. However, they are unsuitably to be used as lubricant due to the undesirable properties of the material. Despite of having dimensional stability of chemical reaction at high temperature, solid lubricants have better performance compared to liquid lubricants in terms of lubricity (Bannister, 1996). Solid lubricants are very desirable for lubrication under extreme condition where the contact surface must be effectively separated while maintaining the lubricant in place. The application often used in aviation industries where high load is applied to sliding surfaces in the presence of boundary and mixed frictional regimes or sometimes at low hydrodynamic speed (Bart et al., 2012a).

Allam (1991) stated that though solid lubricant can still work without cooling system and seal system to separate the fluid between the regimes. However, they face a few drawbacks where the coefficient of friction experienced by solid lubricants is higher than liquid based lubricants. Other than that, most of solid lubricants based are from non-renewable sources. Solid lubricants can be categorised as structural lubricants (graphite and metal dichalcogenides), mechanical (thermoplastics and thermosets), reactive soaps (salts of stearic, oleic and palmitic acid coated with zinc phosphate) and chemically active lubricants (Bart et al., 2012a). Molybdenum disulphide ( $\text{MoS}_2$ ) and graphite are materials that are typically used in solid lubrication. Both materials have good oxidation stability when in high temperature environment due to the presence of crystalline properties (Rudnick, 2009).  $\text{MoS}_2$  and graphite are the earliest compound used in solid lubrication industries because of the laminar structure which makes the shear easier in