



UNIVERSITI TEKNIKAL MALAYSIA MELAKA

**OXIDATION STABILITY COMPARISON BETWEEN ZINC
DIALKYLDITHIOPHOSPHATE AND ZINC
DIAMYLDITHIOCARBAMATE INDUCED PALM OIL
BIOLUBRICANTS**

This report submitted in accordance with requirement of the Universiti Teknikal Malaysia Melaka (UTeM) for the Bachelor's Degree in Technology Automotive (Department of Mechanical Engineering Technology) (Hons.)

by

MUHAMAD NAQIB BIN NADZRI

B071310863

921121085343

FACULTY OF ENGINEERING TECHNOLOGY

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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 Engineering Technology Automotive with Honours. The member of the supervisory is as follow:

.....

MUHAMAD AZWAR BIN AZHARI

ABSTRAK

Perkembangan kesedaran alam sekitar terhadap pencemaran kerana penggunaan minyak mineral yang tidak mesra alam menyebabkan pertimbangan kegunaan minyak baru seperti minyak sayuran. Walaubagaimanapun, minyak sayuran mempunyai kelemahan kestabilan oksida yang menyekat pembangunan minyak baru. Kajian ini menumpukan pada pembangunan minyak-bio baru dengan bahan tambahan ion zink anti-oksida. Minyak kelapa sawit komersial dipilih akan dicampurkan menerusi kaedah percampuran terus ke dalam dua sampel berbeza dengan kepekatan berlainan dari ZDDP dan ZDDC dengan setiapnya pada 1.2 berat %, 1.6 berat % dan 2.0 berat % kepekatan dan dipanaskan selama 20 minit pada 50°C untuk formulasi yang betul. Kandungan logam minyak-bio baru diuji dengan ASTM D6595 dan karakteristik oksida ia dianalisa dengan ASTM D2272. Sifat takat kilat sampel kemudian diuji dengan ASTM D93. Menerusi ujian kandungan logam, ia menunjukkan formulasi yang berjaya untuk 2.0 berat % minyak-bio ZDDP dengan kandungan fosforus pada 791.64ppm dan kandungan zink pada 825.74ppm. Pada 2.0 berat % minyak-bio ZDDC juga menunjukkan formulasi berjaya dengan kandungan fosforus dan zink lebih rendah pada 28.44ppm dan 673.06ppm. Dari ujian kestabilan oksida, ia menunjukkan bahan tambahan anti-oksida menambah baik kestabilan oksida minyak-bio dengan 2.0 berat % ZDDP teroksida pada 128 minit, sementara 2.0 berat % ZDDC teroksida pada 62 minit. Walaubagaimanapun, prestasi minyak mineral didapati lebih baik dalam kestabilan oksida pada 300 minit. Dari ujian takat kilat, prestasi minyak-bio didapati lebih baik berbanding minyak mineral dimana cuma mencapai 204°C, manakala 2.0 berat % ZDDP pada 205°C dan 2.0 berat % ZDDC pada 224°C. Dari kajian pembangunan baru minyak-bio menunjukkan minyak kelapa sawit dengan 2.0 berat % ZDDP mempunyai keputusan yang wajar. Dapat disimpulkan bahawa dengan penambah baikan dalam kestabilan pengoksidaan, minyak-bio kelapa sawit sesuai digunakan sebagai minyak pelincir perindustrian.

ABSTRACT

The environmental concern growth towards pollution due to usage of mineral oils which non-environmental friendly causes consideration of new lubricant usage such as vegetable oils. However, vegetable oils have poor oxidation stability restrain the development of new oil. This study is focusing on developing new bio-lubricant with zinc ion anti-oxidants additives. A commercialized palm oil is chosen were blended through direct introduction methods into two different samples with diversified concentrations of ZDDP and ZDDC with each at 1.2wt%, 1.6wt% and 2.0wt% concentrations and heated for 20minutes in 50°C for proper formulation. The new bio-lubricant metal content were tested with ASTM D6595 and its oxidation characteristics are analyzed with ASTM D2272. The samples flash point properties then tested with ASTM D93. Through the metal content test, it indicates successful formulation for 2.0wt% of ZDDP bio-lubricants with phosphorus content at 791.64ppm and zinc content at 825.74ppm. At 2.0wt% of ZDDC bio-lubricants also shows successful formulation with lower phosphorus and zinc content at 28.44ppm and 673.06ppm. From the oxidation stability test, it indicates that anti-oxidant additives improve bio-lubricant oxidation stability with 2.0wt% of ZDDP degraded at 128 minutes, while 2.0wt% of ZDDC degraded at 62 minutes. However, the mineral oil were found better performance in oxidation stability at 300 minutes. From flash point test, the bio-lubricant were found better in performance compared to mineral oils which only reached 204°C, while 2.0wt% ZDDP at 205°C and 2.0wt% ZDDC at 224°C. From the study of the newly developed bio-lubricant shows palm oil with 2.0 wt% of ZDDP possess the desirable result. It can be concluded that with further improvements in oxidative stability, palm oil bio-lubricant are suitable to be used as industrial lubricant.

DEDICATION

This thesis I dedicated to my beloved family Nadzri Bin Musa and Mariatun Binti Harun. And also to my supervisor, Mr Muhamad Azwar Bin Azhari and my friends for endless support and encouragement to finish my thesis.

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LIST OF ABBREVIATIONS

%	–	Percentage
ADDC	–	Antimony Dialkyldithiocarbamate
API	–	American Petroleum Institute
ASTM	–	American Society for Testing and Materials
B.C	–	Before Christ
°C	–	Degree Celsius
cSt	–	Centistoke
g	–	Gram
H	–	Hydrogen Atom
H*	–	Free Hydrogen
H ₂ O	–	Water
J	–	Joule
K	–	Kelvin
mg	–	milligram
mm	–	millimetre
nD	–	Refractive Index
No.	–	Number
OH	–	Hydroperoxides
P	–	Phosphorus
PAG	–	Poly Alkylene Glycol

PAO	–	Polyalphaolefin
PIB	–	Polyisobutylene
PTFE	–	Polytetrafluorethylene
R	–	Radical
RH	–	Unsaturated Fatty Acid
ROH	–	Alcohol
R*	–	Free Radical
RO*	–	Alkoxy Radical
ROO*	–	Peroxy Radical
ROOH	–	Hydroperoxides
RDE-AES	–	Rotating Disc Electrode Atomic Emission Number
S	–	Sulfur
SAE	–	Society Automotive Engineering
SAE	–	Society of Automotive Engineers
SI	–	System International
VI	–	Viscosity index
wt%	–	Weight percentage
ZDDC	–	Zinc Dialkyldithiocarbamate
ZDDP	–	Zinc Dialkyldithiophosphate
µm	–	micrometre
R^1, R^2, R^3, R^4	–	Alkyl Groups

CHAPTER 1

INTRODUCTION

1.1 Introduction of Lubricant

The principle of supporting a sliding load on a friction reducing film is known as lubrication (Ludema, 1996). The substance of which the film is composed is a lubricant, and to apply it is to lubricate (Nehal et al., 2009). A lubrication substances supply a protective film which allows for two touching surfaces to be unattached, thus reducing the friction between them. Natural lubricant from vegetable oils and animal fats were used until the 19th century (Suhane, 2012).

In the studies of Nehal et al., (2009), they stated that the primary function of lubricants is to lubricate moving parts and carry additives in order to prolong moving parts lifespan. In order to extend the lifespan of moving parts such as an engine, lubricant is used to reduce friction, heat output and wear by creating a liquid film between the textures of a moving parts (Azhari et al., 2015).

Based on Nehal et al., (2011), Lubricant are also used to decrease the rate of oxidation and prevent components from rust. It is also used to supply insulation in transformer utilization. Furthermore, lubricant is used to convert the mechanical power in hydraulic liquid power applications and to seal against contaminants such as dirt, dust and water from entering the parts.

A lubricant are mostly a substance in the form of liquids such as mineral based oils, synthetic based esters, natural based oils, silicone liquids, and water. However, they can be in the form of solids such as polytetrafluoroethylene (PTFE) for the use to

lubricate dry bearings. Grease which is also a lubricant in the form of semi-solid structures, are used to lubricate rotating-element bearings. Besides that, lubricants in the state of gases such as air are used in gas bearings (Hamrock et al., 2004).

An imperative property of lubricants used to improve the friction and wear attributes is the capacity to form persistent antiwear tribofilms firmly attached to the metal surfaces. With legitimately chosen oils that have been fortified, lubricant can overcome the drawbacks of mechanical energy losses in a machine due to high friction and excessive wear which lead to decreased efficiency and shorter machine lifespan (Komvopoulos et al., 2006).

The motives of motor lubrication are to keep away parts from direct contact between the different rolling and sliding components. This is achieved through the arrangement of a thin oil film between the reaching surfaces. Lubricants have the accompanying focal points: Reduction of grinding and wear, dispersal of grinding warmth, delayed bearing life, counteractive action of rust and assurance against harmful aspects (Azhari et al., 2014).

1.2 History

Centuries ago, farmers have been lubricating their ox carts axles with animal fats (Nehal et al., 2009). On other note, research by Azhari et al., (2015) stated that, as early as 1400 B.C., combination of calcium and fats produce grease which then was used to lubricate chariot wheels. During the 1650 BC, lubricant have been used which the lubrication comes from vegetable oils and animal fats, where the Egyptians used animal fats to lubricate wheels chariot. And whale fats are used to lubricate equipment and other machinery (Cheenkachorn et al., 2006).

Since the invention of machines, man have been used lubricants and lubrications to lubricate them (Suhane, 2012). During 16th century, lubricant use is from vegetable oils, animal oils, or mixture of two (Anderson, 1991). Beeswax, animal tallow and water have been used to lubricate wooden cart wheels and bearing (Azhari et al., 2015). In

1845, one of the first usage of the crude oil as a lubricant is from a cotton spinning mill in Pittsburgh, Pennsylvania (Anderson, 1991). It is believed that, to fulfill up-to-date needs, people has been already used lubricant from thousand years ago and it is improved from time to time (Azhari et al., 2015)

In the early of 1769, the automobile history began with the creation of steam engine automobiles that capable to transport human. Various forms of internal combustion engine were developed before the 19th century, but their usage was hindered until the commercial drilling and production of petroleum which began in the mid 1850's. Furthermore, with the enhancement of internal combustion engine, reliable and safer operations of a vehicle at desired operating conditions needs effective lubrication of the moving components to move smoothly over each other. Moreover, lack of lubrication in the engine will cause the engine parts to damage due to heat buildup, rapid wear due to friction and meet its peak and fail, which often takes place at pistons and their cylinders (Suhane, 2012).

During the 19th century, mostly every machines industrials have transformed and changed by the technology to improve the economy and culture situation during the time which is called as Industrial Revolution (Azhari et al., 2015). Since then, the petroleum based oil vastly takes over the field (Anderson, 1991). As the development of larger machines which moves at higher speeds causing the enhancements of lubricant in oil properties. This which led to synthetic lubricant which provides excellent viscosity characteristics thus retaining its viscosity over wide range of temperature (Azhari et al., 2015).

For over a century, mineral based lubricant have dominated lubricant field and in the present time, the environmental issue begin to arise as the mineral based lubricant and synthetic based lubricant is not easily biodegradable. (Nizam et al., 2009). Environmental concern remain to grew due to pollution from excessive lubricant application and disposal, especially total loss lubricants (Erhan et al.,2006).

1.3 Problem Statement

The interest of lubricants turned out to be high a short time later due to quick industrial sector, putting weight on the cost and accessibility of oils from vegetable and animal sources (Suhane, 2012). Since the last decade, there has been a constant demand for unpolluted lubricants due to the strict governments policy and environmental regulations (Adhvaryu et al., 2005). Furthermore, it is also supported by Kržan et al., (2004), mineral oils have a disadvantages of poor biodegradability and has potential of long-term pollution towards the living organisms. Mineral oil based lubricants are seen to be one of the contributor to environmental contamination. This issue happens when the squandered oil created and their consequent transfer cause genuine ecological risks (Azhari et al., 2014).

Mineral based oils are non-friendly lubricant to the environment, non-renewable oils and it is not biodegradable lubricant (Erhan et al., 2006). As most of the lubricants comes from petroleum crude which is toxic to the living organisms and difficult to dispose. Furthermore, vegetable oils with high in oleic acid content are being considered as a potential candidates to replace conventional mineral based lubricants and synthetic esters (Adhvaryu et al., 2005). Other than that, Cheenkachorn et al., (2006) also stated most of lubricant maker have put an effort to reconsider natural oils as base stock due to its biodegradable ability had reached worldwide attention.

During 1650 B.C, lubricants are used based on vegetable oils. In present time, vegetable oils such as palm oil, canola oil, karanja oil, corn oil, and soy oil were being developed to replace mineral based lubricants (Gawrilow, 2004). According to Lawal et al., (2013) the vegetable oils originally have great properties. One of the main factor to be accepted into the market as it is high fluidity, viscosity index, low evaporative loss, high flash point, high biodegradability and low toxicity with regards to their use as base oil for lubricants. Moreover, the vegetable oil have equal or even superior technical performance characteristics to mineral based oils (Kržan et al., 2004).

On the other hand, according to Azhari et al., (2015), the vegetable oils have poor oxidation stability. It creates big problems conveyed by vegetable oil as it causes an

expansion in oil acidity, consistency, erosion and instability keeping in mind the end goal to make them as oil. The high substance of unsaturated fats in vegetable oils creates the oil less cooperative to stabilize the oxidation occurred. When the oil is being used by machine, it oxidizes over a duration of time depending on the type of lubricant, the operational state and the ecosystem. Other than that, oxidation also causes corrosion as it forms water, increased in oil viscosity due to oxidation formation and contributes to wear on the internal surfaces of the machine. Moreover, oxidation can have bad effects on hydraulic and lubrication system, as it will result in sludge and varnish, thus affecting the performance of the equipment. The sludge and varnishes will also accelerates the effect of wear to the components and the temperature gains and accelerate the reaction rates (Anchos et al., 2006).

This oxidation process may be suppressed by using anti-oxidant additives (Azhari et al., 2016). According to Azhari et al., (2015), the addition of Zinc Dialkyldithiophosphate (ZDDP) can slow down the rate of oxidation process in lubricating oil. A study by Dachang et al. (2003), stated that organo zinc compounds such as ZDDP and ZDDC can be used as an anti-oxidizing agent and its also improve anti-oxidation performance in lubricating oil. It is proven that organo zinc compounds will reduce oxidation by hindering the propagation process by reacting with peroxide acting as a peroxide decomposer. ZDDP and ZDDC were identified as organo zinc compound that could be used as peroxide decomposer to reduce the effect of oxidation in lubricating oil (Nehal et al., 2011).

Therefore, this study proposes to compare the additive response zinc ion derivatives which is Zinc Dialkyldithiophosphate (ZDDP) and Zinc Diamyldithiocarbamate (ZDDC) as antioxidant which is rarely being determined.

1.4 Objectives

Based on the problem statement, the objective of this study are stated as below:

- To develop a new bio-lubricant with addition of anti-oxidant agent.
- To test and characterize the new bio-lubricant with standard laboratory test methods
- To compare the new bio-lubricant with mineral based lubricant.

1.5 Work Scope

From the objective stated as above, the work scope for this study have been drawn as below:

- Developing a new biodegradable lubricant using palm oil and ZDDP and ZDDC as anti-oxidation additives.
- Testing the newly developed bio-lubricants using ASTM D6595 and ASTM D2272
- Characterizing the newly developed bio-lubricants in accordance to ASTM D93.
- Comparing the newly developed bio- lubricant with SAE15W-40 mineral based lubricant.

Chapter 2

LITERATURE REVIEW

2.1 Lubricant

During the early age of lubricants usage, liquid form lubricant have been used by people for a great many years prior. The Egyptians demonstrates that the anaglyph record that the general population of mankind utilized the oil (water) to bring the statue. A lubricant is a substance of liquid form in order to prolong machines lifespan by reducing wear and friction in moving by lessen grating between the surfaces in contact with, which eventually diminishes the temperature rise produced during the moving parts surface operation. It might likewise have the capacity of transmitting power, transporting remote particles, or warming or cooling surface. Generally, a substance which produce a film is lubricant and by applying it is to lubricate. In automotive fields, lubricants are used to prolong engine lifespan by reducing temperature rise, eliminate friction and wear limiting from the metal to metal surface contact and also act as a contaminants preventive agents for the vehicle motor (Nehal et al., 2009).

Lubricants are classifieds into three states which divided based on its forms. They are solid form, semi-solid and liquid lubricants. Each of these classifications has its own specific industrial usage. As this study is about liquid lubricants, a further note will be explained based on lubrication regimes and known as Stribeck Curves.