UNIVERSITI TEKNIKAL MALAYSIA MELAKA

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THE ADDITION OF MoDTP IN ZnBuDP INDUCED PALM OIL BIO-LUBRICANT AS TRIBOLOGICAL PROPERTY IMPROVER

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

by

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950709-02-5889

FACULTY OF ENGINEERING TECHNOLOGY

2017

UNIVERSITI TEKNIKAL MALAYSIA MELAKA

BORANG PENGESAHAN STATUS LAPORAN PROJEK SARJANA MUDA

TAJUK: **THE ADDITION OF MoDTP IN ZnBuDP INDUCED PALM OIL BIO-LUBRICANT AS TRIBOLOGICAL PROPERTY IMPROVER**

SESI PENGAJIAN: **2017/18 Semester 1**

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

....

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ABSTRAK

Kajian ini adalah untuk membangunkan pelincir yang baru untuk menangani pengurangan minyak mentah, kenaikan harga minyak dan pencemaran alam sekitar yang disebabkan oleh minyak berasaskan mineral. Pelincir berasaskan minyak sayuran telah dijumpai sebagai pelincir alternatif kerana sifat-sifat biodegradasi, boleh diperbaharui, mesra alam dan tidak mengandungi toksik. Pelincir yang baru dirumuskan menggunakan 2 wt% konsentrasi Zinc Dibutyloctyl dithiophosphate (ZnBuDP) dengan tambahan Molybdenum Dithiophospate (MoDTP) pada kepekatan 0.05 wt% , 0.10 wt%, 0.15 wt% dan 0.20 wt% . Sampel telah diuji mengikut kaedah *American Society for Testing and Material* (ASTM). ASTM D6595 digunakan dalam sampel ujian untuk menentukan kandungan logam sampel yang dikumpulkan oleh Rotating Disc Electrode Atomic Emission Spectroscopy (RDE-AES). ASTM D4712 digunakan dalam pencirian sampel untuk mencirikan geseran pekali dan diameter parut haus oleh empat penguji bola dan mikroskop laser mengikut ujian masing-masing. Pemalar geseran paling rendah direkodkan pada 0.065 manakala diameter parut haus terendah didapati pada 79.14 μm. Keputusan mendapati keputusan yang diingini adalah pada kepekatan 2 wt% ZnBuDP dan 0.05 wt% MoDTP. Kesimpulannya, gabungan ZnBuDP dan MoDTP yang ditambah ke dalam minyak kelapa sawit berjaya dibangunkan dan dirumuskan sebagai bio-pelincir yang baru.

ABSTRACT

This study is to develop a new formulated bio-lubricant. The development of bio-lubricant which is biodegradable, non-toxic and renewable resources are desired since the existing mineral based lubricant are of non-renewable sources and prone to environmental issues. With the increasing concerns of environmental issues, palm oil has been use to replaced mineral oil as lubricant. Zinc Dibutyloctyl Dithiophosphate (ZnBuDP) and Molybdenum Dialkyl Dithiophosphate (MoDTP) was introduced into palm oil to improve the tribological properties in order to make it comparable with commercialized mineral based lubricant. The sample prepared will be added with constant concentration of ZDDP at 2wt% while the concentration of MoDTP varies from 0.00wt% to 0.20wt% with interval 0.05wt% into palm oil and the total weight for each sample bottle is 200g. The samples were tested in accordance to the American Society for Testing and Material (ASTM) method. ASTM D6595 was used in sample testing in determining the metal content of samples collected by Rotating Disc Electrode Atomic Emission Spectroscopy (RDE-AES). ASTM D4712 was used in sample characterization to characterize the coefficient friction and wear scar diameter by four ball tester and upright laser microscope respectively. The lowest coefficient of friction was recorded at 0.065 while the lowest wear scar diameter obtained at 79.14 μm. Result found the desirable results recorded was at concentration of 2 wt% of ZnBuDP and 0.05 wt% of MoDTP. In conclusion, the combination of ZnBuDP and MoDTP induced into palm oil was successfully developed as newly formulated bio-lubricant.

DEDICATION

Special dedication to my beloved family members, especially to my father Khairul Anuar Bin Ahmad and my mother Sa'adah Binti Abd Hamid who always supported and encouraged me with motivation and love through my whole journey.

To my respected and professional supervisor, Mohd Faruq Bin Abdul Latif for endless guidance and support from bottom to the top.

To my helpful friends, Muhammad Bin Otsman and Muhammad Abi Dzar Bin Zainudin, my fellow colleague and all faculty members. For all your care, support and believe in me.

ACKNOWLEDGEMENT

First and foremost, I would like to recite Alhamdulillah and thanks to Allah for providing me strengths and courage to successfully finishing up my thesis. This thesis would not have been possible to complete without the support from many people surround me. I wish to express my sincere appreciation to my beloved supervisor, Mr. Mohd Faruq Bin Abdul Latif for valuable experience, encouragement, guidance, critics and friendship. For the time being in preparing this thesis, I would also like to thank you to all people I was contact, the researchers, the academicians and practitioners which has been contributed in my understanding and thoughts.

I am very thankful to Universiti Teknikal Malaysia Melaka (UTeM) for providing a great facilities in the campus and also to all my the lecturers, tutors and teaching engineers of Faculty of Engineering Technology (FTK) for their support and motivation during this project development, a deep thankfulness for everything and may God bless all of us.

Last but not least, biggest appreciation to my entire family especially my beloved father and mother, Mr. Khairul Anuar Bin Ahmad and Pn. Sa'adah Binti Abd Hamid and family members for their continuous supports from the initial of this project till the end of it. My sincere appreciation also extends to all my fellow friends which is Muhammad Bin Otsman, Muhammad Abi Dzar Bin Zainudin, Muhammad Haziq Ilham Bin Roslan, Muhammad Safwan Bin Rosly and others who have provided assistance at various events and conditions. Their views and tips are useful indeed. Thank you for the time sacrificed to accompany me.

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SYMBOLS, ABBREVIATIONS AND NOMENCLATURE

ASTM	-	American Society for Testing and Materials
COF	-	Coefficient of Friction
C_nH_{2n+2}	-	Saturated hydrocarbon general formula
cSt	-	Centistokes
EP	-	Extreme Pressure
MoS_2	-	Molybdenum disulfide
MoDTP	-	Molybdenum Dithiophosphate
MoDTC	-	Molybdenum dithiocarbamate
PAOs	-	Polyalphaolefin
PIBs	-	Polyisobutylenes
PAGs	-	Polyalkylene glycols
PTFE	-	Polytetrafluoroethylene
RDE-AES	-	Rotating Disc Electrode Atomic Emission Spectroscopy
SAE	-	Society of Automotive Engineers
WSD	-	Wear scar diameter
ZDDC	-	Zinc Dialkyl Dithiocarbamate
ZDDP	-	Zinc Dialkyl Dithiophosphate
ZnBuDP	-	Zinc Dibutyloctyl Dithiophosphate
C	-	Carbon
ml	-	Millilitre
N	-	Newton
S	-	Sulphur
Eq	-	Equation
e.g	-	Example

g	-	Gram
F	-	Fahrenheit
m	-	Meter
mm	-	Millimeter
mg	-	Milligram
%	-	Percent
℃	-	Degree of Celcius
μ	-	Coefficient of Friction
wt%	-	Weight percent
μm	-	Micrometer
mm^2/s	-	Kinematic Viscosity (millimetre square per second)
C	-	Carbon
H	-	Hydrogen
KOH	-	Potassium Hydroxide
Mo	-	Molybdenum
MoS_2	-	Molybdenum Disulphide
OH	-	Hydroxide
R	-	Radical
S	-	Sulphur
ROOH	-	Organic Acid

CHAPTER 1

INTRODUCTION

1.1 Introduction of Lubricant

Lubricant oil is already used by human since ancient times. During 1400 BC, grease made of a combination of calcium and fat from animal were used to lubricate chariot axels (Azhari et al., 2015c). After the discovery of petroleum, grease with the blending of potassium, calcium, and sodium soaps were placed on the market in limited quantities (Pirro and Wessol, 2001). Since 1650BC, natural oil was used as a lubricant. This natural lubricant gathered from natural resources such as olive, rapeseed, castor beans, palm oil, and the fats from sperm whale, animal lard, and wool (Gawrilow, 2004). It is believed that, lubricant has been used by people long time ago and it has upgraded a long time to time to achieve today's needs.

In all types of machines, the surfaces of moving or sliding or rolling parts rub against each other. Due to the mutual rubbing of one part against another, a resistance is offered to their movement. This resistance is known as friction. It causes a lot of wear and tear of surfaces of moving parts. Any substance introduced between two moving or sliding surfaces with a view to reduce the friction or frictional resistance between them, is known as a lubricant (Bannister, 1996). The main purpose of a lubricant is to keep the moving or sliding surfaces apart, so that friction and consequent destruction of material is minimized. The process of reducing friction between moving or sliding surfaces, by the introduction of lubricants in between them, is called lubrication (Holweger, 2013).

Lubricants reduce wear and tear of the surfaces by avoiding direct metal to metal contact between the rubbing surfaces (Abdullah et al, 2013). Due to frictional heat and destruction of material, lubricant reduces expansion of metals. It also acts as a coolant of metal due to heat transfer media to protect metal surfaces against corrosion, to flush away or prevent the ingress of pollutants and to keep the mating component reasonably free from deposits. It avoids unsmooth relative motion. (Sharma and Gandhi, 2008).

Lubricants can be classified based on the following criterions which are solid lubricant, semi-solid lubricant and liquid lubricant (Mortier and Orszulik, 2012). Solid lubricant is the film of a solid material which composed of inorganic or organic compounds, such as graphite, molybdenum disulphide, and cadmium disulphide. Semi solid lubricant is liquid suspended in a solid matrix of thickener and additives, such as grease. Liquid lubricant is oils such as petroleum, vegetable, animal, and synthetic oils. (Mobarak et al., 2014).

There are three base oil resources which are natural oil, refined oil, and synthetic oil. Natural oil determined that the oils were derived from animal fats and vegetable oil. Refined oil means that the oils were derived from crude oil or petroleum reserves such as paraffinic, naphthenic, and aromatic oils. Synthetic oils are oils synthesized as end products of reactions that are tailored per requirement examples are synthetic esters, silicones, and polyalphaolefines (Mobarak et al., 2014).

1.2 Potential of Vegetable Oil as Lubricant

Nizam and Abdul Bari, (2009) stated that the vegetable oil gains popularity because of the advantage to the environment. As an alternative lubricant source, lubricants that were formulated from vegetable oils show better performance than mineral oil based lubricants in properties such as lubricity leading to reduced wear and friction, higher viscosity indices and elevated flash points. The lubricants from vegetable oil are safer for use (Ponnekanti and Savita, 2012).

Vegetable oil can be obtained from oil-containing seeds that are available through-out the world (Aluyor and Ori-Jesu, 2008). Vegetable oil can both be edible and non- edible. An example of vegetable oil includes jatropha, karanja, neem, rice bran, rapeseed, castor, linseed, mahua, palm, sunflower, coconut, soybean, olive, and canola (Mobarak et al., 2014). The lubricant from non-edible vegetable oil feedstock can overcome the economic, environmental and food versus fuel issues associated with edible vegetable oils (Gui et al., 2008).

Vegetable oils can be used as a lubricant in their natural form. There are several advantages and disadvantages when considered for industrial and machinery lubrication. Vegetable oils are biodegradable and non-toxic and renewable resources (Randles and Wright, 1992). It also low in volatility due to the high molecular weight of the triglyceride molecule and excellent temperature viscosity properties. Their polar ester groups are able to adhere to metal surfaces, and therefore, possess good lubricity. In addition, vegetable oils have high solubilizing power for polar contaminants and additive molecules. According Aluyor and Ori-Jesu, (2008) stated that, the use of vegetable oils include their relatively low viscosity-temperature variation, which is their high viscosity indices, which are about twice those of mineral oils.

On the other hand, vegetable oils in their natural form lack sufficient oxidative stability for lubrication application. Vegetable oils also have low-temperature limitations, unpleasant smell, poor compatibility with paints and sealants, flushing propensity because of low viscosity, and filter-clogging tendency.

According Azhari et al., (2015b) stated that, adding Zinc Dialkyldithiophosphate (ZDDP) in vegetable oils is one of the solutions to overcome the oxidation. Therefore, ZDDP has an ability to prevent wear and it was used for their antioxidant properties. Zinc Dialkyldithiophosphate (ZDDP) can be a solution to overcome the oxidation because ZDDP has an outstanding performance as antioxidant agent and ZDDP also has abilities as a radical scavenger and hydroperoxide decomposition (Azhari et al., 2015c). A study by Azhari et al., (2015b) stated that, better performance could be achieved if the addition of ZDDP as additive in lubricant and ZDDP can act as an improver of physical properties in certain vegetable oil and other additives compare to vegetable oil without ZDDP.

1.3 Problem Statement

Mineral oil, synthetic oil, refined oil and vegetable oil are available over the world. Mineral oil which are derived from petroleum oil is widely used but not adaptable to the environment because of their toxicity and non-biodegradability (Adhvaryu et al., 2005; Salih et al., 2012). The reduction of the world's crude oil reserve added with the consumption rate, increase in petroleum prices, and issues related to conservation have brought about renewed interest in the use of bio-based materials.

The petroleum based lubricants can pollute the air due to its volatility. This pollution is hazardous not only plants and animals inhabiting the contaminated areas, but potentially human residents as well. Mineral oil is known to be non-renewable, non-biodegradable and toxic because it was obtained from crude oil sources and also harmful to the environment. It also can affect human life (Bilal et al., 2013). Emphasis on the development of renewable, biodegradable, and environmentally friendly industrial fluids, such as diesel, lubricants and other fuels have elevated the need to search for alternative renewable sources as lubricant (Sahoo et al., 2007; Basha et al., 2009; Demirbas, 2009; Refaat, 2010).

The use of vegetable oil-based lubricants can reduce both reliance on petroleum and anthropogenic impact on the environment. This product is call bio-lubricants that carry several environmental, health, and performance benefits over petroleum based lubricant (McNutt, 2016). Vegetable oil is a vital role to substitute the petroleum based lubricant partially because it has numerous advantages over base lubricant which are renewability, environmentally friendly, biodegradability, and less toxicity (Ing et al., 2011; Shahabuddin et al., 2012b; Salunkhe, 1992).

Vegetable oils are mainly triglycerides. Triglycerides contain three hydroxyl groups and long chain unsaturated free fatty acids attached at the hydroxyl group by ester linkages (Fox and Stachowiak, 2007; Jayadas and Nair, 2006). The unsaturated free fatty acid is the ratio and position of a single bond, double bond, triple bond of carbon chain which is oleic, linoleic, and linolenic fatty acid (Waleska et al., 2005).

Vegetable oil based lubricant has some disadvantages which are high viscosity at low temperature and poor oxidative stability at high temperature makes it easily to oxidize. Vegetable oils without additives could make the vegetable oils out-performed mineral based oils in anti-wear and friction, scuffing load capacity, and fatigue resistance. Means it cannot reduce friction and wear effectively. (Syahrullail et al., 2013). According Tiong et al., (2012), vegetable oil becomes less effective under extreme loads that show the vegetable oils are principally effective as boundary lubricants. To overcome these problems, additives are designed specifically to eliminate the problems of friction and wear which is ZDDP and MoDTP.

Additions of ZDDP in vegetable oil can be suitable additives because it will enhance the formation of the film to prevent wear (Burkinshaw, 2014). ZDDP has limited water solubility, low vapour pressure, high viscosity and partition preferentially into the hydrophobic (oil) phase. ZDDP act as anti-wear and the fundamental mechanism of ZDDP is the reaction of ZDDP with the metal surface to form a solid protective film and the reaction layer. When metal is submerged in ZDDP solution in a lubricant or other non-polar solvent, a thermal film rapidly forms at the metal surface (Azhari et al., 2015b). ZDDP also has limitations which are on concentration. If the concentration is too high it will increase the thickness of film and decrease the performance as anti-wear.

Molybdenum compounds also can be suitable additive induced vegetable oil because it can be used individually in mineral based oil to reduce friction and wear (Tang and Li, 2014). Molybdenum containing compounds has verified as modifier additives for engine oils through the formation of decomposition of MoS_2. This is done by complex tribochemical reaction to reduce friction (Azhari et al., 2016).

There are two names of molybdenum compound which is Molybdenum dithiocarbomate (MoDTC) and Molybdenum dithiophosphate (MoDTP). Both also acting as modifier additives to reduce friction and wear. Molybdenum dithiocarbomate (MoDTC) is an excellent anti-friction agent because it can easily break its bond (Liskiewickz et al., 2013). The sheets MoS_2 will generate from decomposition of MoDTC. Formation of MoS_2 leans more towards pure sliding conditions compared to sliding or rolling conditions. The amounts of molybdenum containing additives depend on the formation of MoS_2 at asperity contacts when low friction is produced.

It is expected that, molybdenum compounds when added with Zinc Dialkyldithiophosphate (ZDDP) will form a better anti-wear performance (Azhari et al., 2015a). However, the mixing concentration of these two compounds is still not established.

As such, this study will propose to use palm oil with the addition of zinc dibutyloctyl dithiophosphate (ZnBuDP) and Molybdenum dithiophosphate (MoDTP) which is rarely being studied by another researcher.

1.4 Objective

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

1. To develop a new bio-lubricant oil with the addition of friction modifier and anti-wear agent.
2. To test and characterize the newly developed bio-lubricant.
3. To compare the tribological performance of newly developed bio-lubricant with existing mineral based oil.

1.5 Scopes

In order to reach the objectives, a few scopes have been drawn:

1. Developing new bio-lubricant oil using ZnBuDP and MoDTP induced palm oil.
2. Testing of the newly developed oil using standard laboratory test methods (ASTM).
3. Characterizing of the newly developed oil by conducting four-ball test.
4. Comparing the newly bio-lubricant with existing mineral based oil.

CHAPTER 2

LITERATURE REVIEW

2.1 K-chart

K-chart is presented in the form of a tree diagram as depicted in Figure 2.1. There consists of issues, methodologies, results and a time line. K-chart was developed for researcher to easily organize, planning and monitoring the relevant chapter and issues in order to reach the objective of this study (Abdullah et al., 2006).

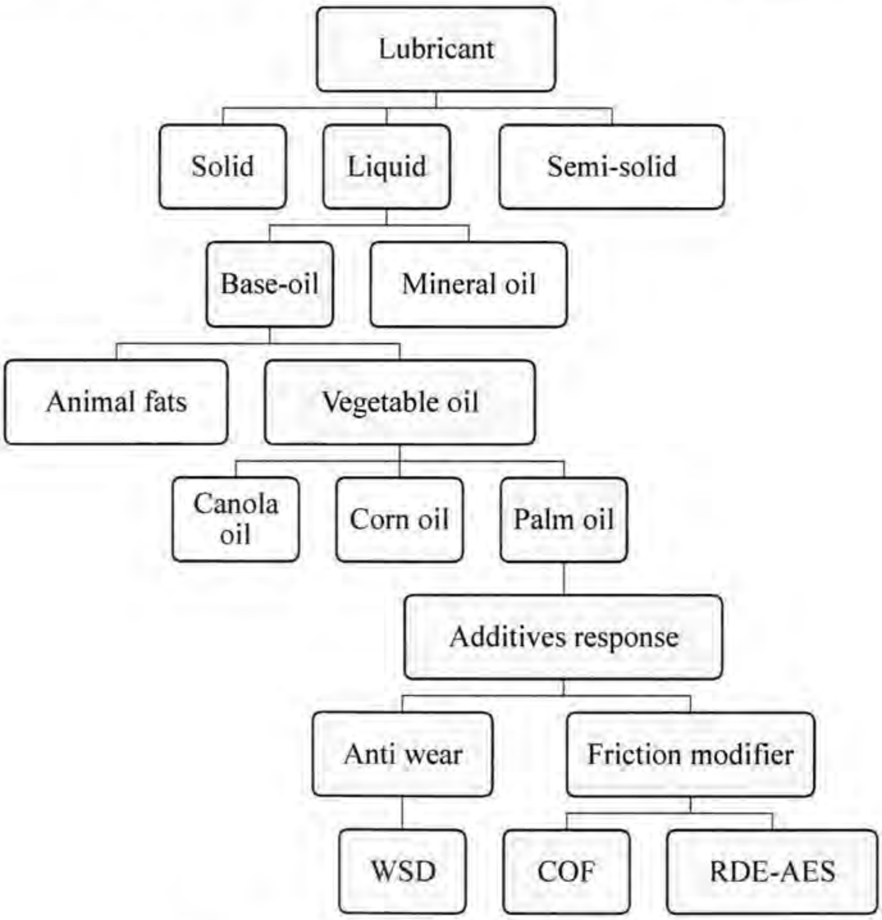

Figure 2.1: Structure of K-chart research flow

2.2 Classification of Lubricant

Lubricants are substances that are used to facilitate two moving parts in contacting surfaces. Lubricants also used as a friction reducer, reducing wear agent and dissipating friction heat (Azhari et al., 2016). Maleque et al., (2003) stated that lubricant may be used to reduce the frictional force, the amount of wear, and the degree of surface adhesion. Lubricants implement a multiplicity of functions in engines and machines, such as it protects metal surfaces against corrosion, acts as a heat transfer agent, and flushes out contaminants (Maleque et al., 2003). Lubricants can be classified into three groups which are solid lubricant, semi-solid lubricant and liquid lubricant (Zhou and Vincent, 1999). Characteristics for good lubrication is high volatility, high boiling point thermal stability, low freezing point, corrosion prevention capability and high resistance to oxidation (Mobarak et al., 2014).

2.2.1 Solid Lubricant

Solid lubricants are widely used for process with oscillatory motion (Muller, 1975). Solid lubricants also are used at very high temperature or under very high radiation that cannot be lubricated by liquid lubricant or greases (Ahmed and Nassar, 2011). Solid lubricants protect counter surfaces from too much wear, reduce noise and vibration induced by stick-slip at the friction interface (Cho et al., 2006). The main purpose of solid lubricants is to build up a continuous adherent soft or hard film in rubbing surfaces (Bartz, 2013). Solid lubricants form a film on the contacting surfaces and substitute the elevated friction of the metal junction for the shear of the solid lubricants film (Muller, 1975).

There are three general types of solid lubricants film which is burnished, bonded, and vacuum-deposited (Hilton and Fleischauer, 1992). Burnished films were produced by a rubbing process transfer solid lubricant onto the contact surface (Rapoport et al., 2009). The tribological properties of burnished films were showed low friction and wear for ceramic materials but these films have low longetivity which is low adhesion, density, and thickness of burnishing by cloth (Moshkovish et al., 2007). Burnishing of Molybdenum disulphide (MoS_2) or other solid lubricants is widely used to improve the tribological properties of roughened substrates (Rapoport et al., 2009). Cloth burnishing and ball burnishing are simple methods for formation of solid lubricant films. The reasons of these two-stage burnishing are to increase the film's thickness (Moshkovish et al., 2007).

Solid lubricants are also used for commercial friction materials which are graphite, molybdenum disulphide (M_oS_2), antimony trisulphide, copper sulphide, calcium fluoride, and others (Cho et al., 2006). Mineral oils and greases are used as additives in order to increase the load carrying capacity of the lubricant. There are three general categories which is lamellar solids, soft metals and polymers (Roberts, 1990). Solid film lubrication can be affected be essentially one of two ways. It can be transferred by rubbing from a solid made from, or containing the dry lubricant. It can be applied to one counter face in the form of film (Torbacke et al., 2013).

Solid lubricant film based on MoS_2 and WS_2 are also widely used for dry air friction and aerospace applications (Moshkovich et al., 2007). Graphite is the most commonly used of all the solid lubricants and can be used either in the powdered form or in suspension. The characteristics of graphite are, non-inflammable and can stable up to a temperature of 375°C (Ludema et al., 1996). The layers of graphite sheets are arranged one above the other and held together by weak van der Waal's forces. These parallel layers which can easily slide one over other make graphite an effective lubricant and the layer of graphite has a tendency to absorb oil and to be wetted of it.

Zhou and Vincent, (1999) stated that MoS_2 is a good lubricant characterized by its hexagonal structure. Molybdenum disulphide has a sandwich like structure with a layer of molybdenum atom in between two layers of sulphur atoms. Poor interlaminar attraction help these layers to slide over one another easily. It is stable up to a temperature of 400°C (Ludema et al., 1996). Molybdenum disulphide (MoS_2) also is an excellent anti-friction agent because it easily brakes its bond. MoS_2 formation lean more towards pure sliding conditions compared to sliding or rolling conditions. The amounts of molybdenum containing additives depend on the formation of MoS_2 at harshness contacts (Azhari et al., 2015c).

2.2.2 Semi-Solid Lubricant

Lubricating oils are used for lubricating moving parts of engines and machines. Grease, which is a semi-solid belongs to this group. Grease are used to reduce friction between two solid surfaces (Udonne, 2011). Basically, there are several types of grease which is clay grease, asphaltic-type grease, extreme pressure grease, soap thickened mineral oils, multi-purpose grease and many more (Abdulbari et al., 2011).

Lubricating grease has been used for plenty of application for efficiency and to prolong the life-span of the machinery (Pogosian and Martiroshan, 2007). Grease can act as a seal, provide protection against corrosion and at the same time being able to reduce noise and shock (Abdulbari et al., 2011). Basically, grease formulation is made from petroleum and it derivatives but this formulation has created environmental pollution due to the nature of non-biodegradability of this base oil (Gui et al., 2008). The application of grease from natural sources is more sought-after.

Spent bleaching earth (SBE) is suitable characteristics to be used as a thickener in grease application. SBE is derived from natural clay. Waste cooking oil also has good characteristics as bio-degradable, cheap, possesses inherent viscosity, higher flash and fire point than mineral oil and provides environmentally friendly solution for grease application (Abdulbari et al., 2011). The thickeners consist primarily of special soaps of Li, Na, Ca, Ba, Al, and others. Non-soap thickeners include carbon black, silica gel, polyureas and other synthetic polymers, clays, and others (Bhushan, 2013). Grease can support much heavier load at lower speed. Internal resistance of grease is much higher than that of lubricating oils. Therefore it is better to use oil instead of