

**TRIBOLOGICAL PERFORMANCE OF EXPANDED GRAPHITE AS OIL  
LUBRICATING ADDITIVE**

**MUHAMMAD YUSUF HIDAYAT**

**This report is submitted  
in fulfillment of the requirement for the degree of  
Bachelor of Mechanical Engineering**

**Faculty of Mechanical Engineering**

**UNIVERSITI TEKNIKAL MALAYSIA MELAKA**

**MAY 2019**

## DECLARATION

I'm Muhammad Yusuf Hidayat declares that this project report entitled "Tribological Performance Of Expanded Graphite As Oil Lubricating Additive", is the result of my own work except as cited in the references

Signature : .....

Name : Muhammad Yusuf Hidayat

Date : .....

## APPROVAL

I hereby declare that I have read this project report and in my opinion this report is sufficient in terms of scope and quality for the award of the degree of Bachelor of Mechanical Engineering (Hons).

Signature                    : .....  
Name of Supervisor : .....  
Date                         : .....

## **DEDICATION**

To my beloved mother and father

## ABSTRACT

This study is focusing on the effect of mineral engine oil with Expanded Graphite (EG) nanoparticles as an additive. The optimal composition of additive added into mineral oil is about 0.5% volume and it is dispersing by using a sonication technique. Then, the most optimal temperature in the Coefficient of Friction is at 75°C. The tools for the tribological testing is Four Ball Tester according ASTM D-4172 standard. The volume loss of ball is analyzing to get the value of wear volume between the engine oil with additives and non-additives. Minimum value of wear volume is found at 0% and 0.5%, both of these show the smallest wear volume in base engine oil when compared to 0.2% EG. For coefficient of friction in term of sliding time. At 75°C, engine oil with 0.5% EG has the best coefficient of friction at a stable engine running condition when compared to all engine oils against temperatures of 40°C and 120°C. Then, Wear scar observation on 0% and 0.5% EG at temperature 75°C. They show different wear patterns with parallel grooves. 0.5% EG has shallow scratches when compared to 0% EG which has more deep grooves. The result of the experimental studies has shown a potential and effective way in reducing friction and wear at the most optimum temperature by apply EG as an additive in the lubricant oil.

## **ABSTRAK**

*Kajian ini memberi tumpuan kepada kesan minyak enjin mineral dengan nanopartikel yang diperluas Grafit (EG) sebagai aditif. Komposisi aditif yang ditambah ke dalam minyak mineral adalah kira-kira 0.5% dan menyebarkan menggunakan teknik sonication. Kemudian, suhu yang paling optimum dalam Peleki Gesekan adalah pada 75 ° C. Alat-alat untuk ujian tribological adalah Four Ball Tester menurut standard ASTM D-4172. Kehilangan jumlah bola menganalisis untuk mendapatkan nilai volume pakai antara minyak enjin dengan bahan tambahan dan bahan tambahan. Nilai minima voltan pakai didapati pada 0% dan 0.5%, kedua-duanya menunjukkan volum haus terkecil dalam minyak enjin asas apabila dibandingkan dengan 0.2% EG. Untuk peleki geseran dari segi masa gelongsor. Pada 75 ° C, minyak enjin dengan 0.5% EG mempunyai koefisien geseran terbaik pada keadaan enjin yang stabil apabila dibandingkan dengan semua minyak enjin terhadap suhu 40 ° C dan 120 ° C. Kemudian, Perhatikan luka pada 0% dan 0.5% EG pada suhu 75 ° C. Mereka menunjukkan corak memakai yang berbeza dengan alur selari. 0.5% EG mempunyai calar cetek apabila dibandingkan dengan 0% EG yang mempunyai alur yang lebih dalam. Hasil kajian eksperimen menunjukkan cara yang berpotensi dan berkesan dalam mengurangkan geseran dan dipakai pada suhu yang paling optimum dengan memohon EG sebagai tambahan dalam pelincir minyak.*

## **ACKNOWLEDGEMENT**

First of all, Alhamdulillah and gratefulness to Allah the Al-Mighty for his kindness that helps me to do this report until done.

I wish to give a lot of thanks to Technical University of Malaysia Melaka (UTeM) for giving opportunity and chances to do and undergo these “Final Year Project”

I wish to acknowledge with many thanks to the most important person who is my supervisor, Dr. Mohd Rody bin Mohamad Zin for his guidance and all of the good advice during the preparation for Final Year Project. Besides that, I would like to give a lot of thanks to all the lecturer and technicians Faculty of Mechanical Engineering for their guidance, opinions and also advices.

I would like to give thanks to my best friend for their guidance, advice and knowledge exchange. There are Dea, Laila, Adam, Ina, Irfan. Lastly, a lot of thanks to my parents and whole family that support me during this Final Year Project.

## TABLE OF CONTENT

<b>DECLARATION .....</b>	<b>ii</b>
<b>APPROVAL .....</b>	<b>iii</b>
<b>DEDICATION .....</b>	<b>iv</b>
<b>ABSTRACT .....</b>	<b>v</b>
<b>ACKNOWLEDGEMENT .....</b>	<b>vii</b>
<b>TABLE OF CONTENT .....</b>	<b>viii</b>
<b>LIST OF TABLES.....</b>	<b>xii</b>
<b>LIST OF FIGURES.....</b>	<b>xiii</b>
<b>LIST OF ABBEREVATIONS.....</b>	<b>xv</b>
<b>LIST OF SYMBOL .....</b>	<b>xvi</b>
<b>CHAPTER 1.....</b>	<b>1</b>
<b>INTRODUCTION .....</b>	<b>1</b>
1.1 Background	1
1.2 Problem Statement	3
1.3 Objective	3
1.4 Scope of Project	3
<b>CHAPTER 2.....</b>	<b>5</b>
<b>LITERATURE REVIEW .....</b>	<b>5</b>
2.1 Lubrication Theory	5
2.2 Type of lubricants	6



2.2.1	Liquid lubricant .....	6
2.2.2	Gas Lubricant .....	7
2.2.3	Solid Lubricant .....	8
2.2.4	Semi Solid Lubricant .....	10
2.3	Wear Mode .....	10
2.3.1	Abrasive Wear .....	11
2.3.2	Adhesive Wear .....	11
2.3.3	Corrosive Wear.....	11
2.3.4	Fatigue Wear.....	11
2.4	Typical Stages of Wear .....	12
2.4.1	Initial Wear Stage .....	12
2.4.2	Steady State Wear Stage.....	12
2.4.3	Catastrophic Wear Stage .....	13
2.5	Nanoparticle Additives .....	13
2.6	Nanoparticle Lubrication Mechanism .....	13
2.6.1	Mending Effect.....	14
2.6.2	Rolling Effect .....	14
2.6.3	Protective Film Formation.....	14
2.6.4	Polishing Effect .....	14
2.7	Graphene .....	15
2.7.1	Few-Layer Graphene (FLG).....	16
2.7.2	Multi-Layer Graphene (MLG) .....	16
2.7.3	Graphene Nanoplatelets (GNP).....	16

2.7.4	Reduced Graphene Oxide (rGO) .....	16
2.7.5	Graphene Oxide (GO) .....	16
2.7.6	Expanded Graphite (EG) .....	17
2.8	Expanded Graphite (EG) on Tribological Aspect .....	17
<b>CHAPTER 3.....</b>		<b>19</b>
<b>METHODOLOGY .....</b>		<b>19</b>
3.1	Introduction .....	19
3.2	General Process .....	20
3.3	Experimental Methodology .....	23
3.3.1	Test Equipment and Specimens.....	23
3.3.2	Raw Material Preparation.....	23
3.3.2.1	Material Selection .....	24
3.3.3	Test Parameter .....	25
3.3.4	Experimental Procedure .....	27
3.3.4.1	Ultrasonic Homogenizer .....	27
3.3.4.2	Ultrasonic Bath .....	28
3.3.4.3	Ducom Four-Ball Tester.....	30
3.3.4.4	Weighing Balance .....	31
3.3.4.5	Kinematic Viscosity Apparatus.....	32
3.3.5	Surface Analysis .....	34
<b>CHAPTER 4.....</b>		<b>35</b>
<b>RESULTS AND ANALYSIS .....</b>		<b>35</b>
4.1	Coefficient of Friction .....	35

4.2	Coefficient of Friction at a stable region	38
4.3	Coefficient of Friction at Running in Region	39
4.4	Wear Scar Diameter	41
4.5	Wear volume $k$ against Different Temperature, °C	43
4.6	Wear scar observation on 0% and 0.5% EG at temperature 75°C	44
<b>CHAPTER 5.....</b>		<b>48</b>
<b>CONCLUSION AND RECOMMENDATION.....</b>		<b>48</b>
5.1	Conclusion	48
5.2	Recommendation	49
<b>REFERENCE .....</b>		<b>50</b>

## LIST OF TABLES

<b>TABLE</b>	<b>TITLE</b>	<b>PAGE</b>
2.1	Properties of Graphene	14
3.1	Physical Properties of Base Oils	22
3.2	Wear Test Conditions	25
4.1	Wear Scar Diameter in mm	42
4.2	Wear Volume for 0% EG, 0.2% EG and 0.5% EG	44

## LIST OF FIGURES

FIGURE	TITLE	PAGE
2.1	Lubrication between two surfaces in contact	5
2.2	Layers lattice structures	8
2.3	Organic compounds like thermoplastic and thermoset	8
2.4	Example of chemical properties in palmitic acid	8
2.5	Illustration of (a) adhesive wear (b) abrasive wear, (c) fatigue, (d) corrosive	9
2.6	Illustration of wear stage	11
2.7	Illustration of lubrication mechanism	13
2.8	Expanded Graphite	16
2.9	Tribological trend of the loads effect on the friction coefficient	17
3.1	Flow Chart for FYP	19
3.2	Gantt Chart for PSM 1	21
3.3	Gantt Chart for PSM 2	21
3.4	0.2% EG additives in mineral oil	23
3.5	Ultrasonic Homogenizer	26
3.6	Ultrasonic Bath	27
3.7	Four-Ball Tester Machine	29
3.8	Weighing Balances	30
3.9	Kinematic Viscosity Apparatus	32
4.1	Coefficient of friction against sliding time at 40°C, 75°C and 120°C for different percentages of expanded graphite in Shell Rimula base oil (a)	35

	SAE 15W-40 + 0% EG (b) SAE 15W-40 + 0.2% EG and (c) SAE 15W-40 + 0.5% EG	
4.2	Graph of Coefficient of friction against different temperature at stable region	41
4.3	Coefficient of friction against sliding time with temperature for different percentage of EG at (a) 40°C (b) 75°C and (c) 120°C	38
4.4	Images of steel ball wear scar	42
4.5	Wear Scar Diameter of steel ball at different temperature and percentage of expanded graphite in base engine oil	43
4.6	Graph of Wear Volume against Temperature	44
4.7	(a) SEM images of the wear scars at an applied load of 392N on the ball specimen	45
	(b) SEM spectra at spots specified in 392 N load in Mineral base oil with 0% EG additives	
	(c) SEM spectra at spots specified in 392 N load in Mineral base oil with 0.5% EG additives	

## LIST OF ABBREVIATIONS

EG	Expanded Graphite
ASTM	American Standard for Testing and Materials
SEM	Scanning Electron Microscope
CuO	Copper Oxide
TiO <sub>2</sub>	Titanium Dioxide
SF	Sulphur Free
NBR	Acrylonitrile-Butadiene Rubber
FLG	Few Layer Graphene
MLG	Multi-Layer Graphene
GNP	Graphene Nanoplatelets
rGO	Reduced Graphene Oxide
GO	Graphene Oxide
2D	Two-Dimensional
SAE	Society of Automotive Engineers
UTeM	University of Technical Malaysia Melaka
SEM	Scanning Electron Microscope
COF	Coefficient of Friction
EDX	Energy Dispersive X-ray
WSD	Wear Scar Diameter
TBN	Total Base Number
PC	Personal Computer
AISI	American Iron and Steel Institute

## LIST OF SYMBOL

TPa	=	Terapascal
kg	=	Kilogram
g	=	Gram
RM	=	Malaysian Ringgit
ml	=	Milliliter
L	=	Liter
N	=	Newton
m	=	Meter
cm	=	Centimeter
mm	=	Millimeter
μm	=	Micrometer
Nm	=	Newton Meter
W	=	Watt
v	=	Volt
s	=	Second
HRC	=	Hardness Rockwell Scale
rpm	=	Rotation Per Minute
/	=	Per
K	=	Kelvin
°C	=	Degree Celsius
%	=	Percent



# CHAPTER 1

## INTRODUCTION

### 1.1 Background

Graphene is a new carbon nanomaterial used worldwide due to its amazing properties such as high surface area, high thermal conductivity, affluent material and high performance with low quantity of material [1]. Despite the fact that graphene requires many kind of studies, Expanded Graphite (EG) which is a highly important form of graphene is examined as a major applicant material used in mineral-based lubricant because of its properties of easy shear capability. Furthermore, graphene plays a very important part in reducing frictional behaviour and improving anti wear ability at optimal concentration when added to paraffin oil [1]. It is a great benefit in the automotive sector for the performance in strengthen, lighten and reduce noise of internal combustion engine such as piston assembly, valve train and bearings. Combination between engine oil with additive oil helps improve in lowering the mechanical loses by 10% to reduce fuel consumption [2]. Graphene is an individual layer of graphite which is used in forming a tribofilm or boundary between sliding contact interfaces and create high shield coating that prevents from corrosion and rusting. Based on previous statement, a good expectation can be obtain if graphite and mineral oil is blended together because the properties of graphite will intensify the lubrication effect. However, the disadvantages of this method is the price of graphite which cost RM 450 per 100 ml. It contributes 33-40 % of tribological properties including the study and application

of fundamental friction, lubrication and wear. Thus, it is shown that it is a serious issue to investigate on performance material of expanded graphite in Mineral Oil.

From previous research, the viscosity, thermal conductivity and flash point can be improved by nanoadditives dispersing. It also increase technical properties of the main component and pour point dramatically [2]. Therefore studies on the performance of expanded graphite (EG) in a mineral oil-based product has become a famous and critical issue. Base mineral oil when blended with nanoparticle improved the tribological characteristic by composition, shape, concentration, grain size and dispersion stabilization [2]. Therefore, the addition of different concentration of EGs should be able to enhance the tribological properties.

Utilizing wear by variation of temperature with same critical load method, the tribological performance of additive in mineral-based lubricant can be tested by using four-ball testing machine. Result can be observe from the size of the wear scar, optimum additive percentage and operating temperature. The four-ball test is one of the techniques in analysing reactions between solid contact and it is effective to improve anti-friction behaviour, anti-wear characteristic and extreme pressure properties of lubricants. Based on the result, the test method can identify not only the property of lubricant but also the additional lubricating particle such as graphene.

## **1.2 Problem Statement**

In recent years, lubrication is a very important material asset in a mechanical system [3]. Lubricant is used in engine to develop better performance as the engine runs. The performance of a lubricant extremely depends on the additives it requires. Mineral oil based lubricant has high friction coefficient and energy losses due to friction especially in boundary regime or high load. Adding the additive nanoparticles could prevent high contact pressure between the friction surfaces. Thus, it is expected to improve wear and friction coefficient in boundary region and also extend the lifespan of an engine.

Therefore, this study investigates the expanded graphite concentration percentage in mineral oil with expectation of improving the tribological properties. Furthermore, this study will be interesting if the optimum result is gain at normal engine running temperature which is 75°C. This is because overall life cycle of an engine are in this state. To achieve the objective of the study, several methods will be used such as analysis of wear mode, wear scar diameter and steel ball profile.

## **1.3 Objective**

The main objectives of this research are as follows:

- i. To study the new potential improvements for friction and wear of Expanded Graphite (EG) in mineral based lubricant.
- ii. To determine the optimum percentage of Expanded Graphite in Mineral based lubricant for friction and wear of steel and steel contact.

## **1.4 Scope of Project**

The scopes that focuses on this project are:

- i. Study the varying concentration of EG with varying of temperatures.
- ii. Test using 4-ball Tester under ASTM D 4172 standard with parameter speed 600 rpm for 1 hour with load 392N.
- iii. Test at 40°C, 75°C and 120°C with load 392N.
- iv. Analyze the result using Scanning Electron Microscope (SEM) to observe the wear morphology.

## CHAPTER 2

### LITERATURE REVIEW

#### 2.1 Lubrication Theory

Nowadays, lubricants are usually introduced as a substance between two moving surfaces to reduce friction and wear properties. Lubricants act as protective film that coats and protects two contact surfaces. It also smoothens out the surface by filling up the gaps of the rough asperities [4] as well as dissolving minute particles and distributing heat. Figure 2.1 shows the presence of lubrication between two surfaces in contact. Furthermore, lubricants can be used to improve the oxidation resistance which helps prevent rust and corrosion. It can also be used to control and dissolve contamination such as reaction products, wear particles and other debris. When two surfaces are moving, heat is generated due to friction. An application of a protective film in between the moving surface will reduce friction and heat generation. Thus, achieving wear reduction.

Based on previous research in various lubrication systems including gas lubrication, solid lubrication, and liquid lubrication, early civilisations used gypsum, water lubrication, and animal fat as a method to reduce friction [5]. Then, civilisations within Middle East began incorporating oil as a method of lubrication, which then made it as part as their culture and tradition [5]. This inclusion of lubrication as part of society existed centuries prior to the invention of mechanical engines. Lubricant application in daily life started from the mid-1400s, and it was only starting in the mid-17th century that petroleum-based lubricants were recognised for the reduction of friction and wear [6].

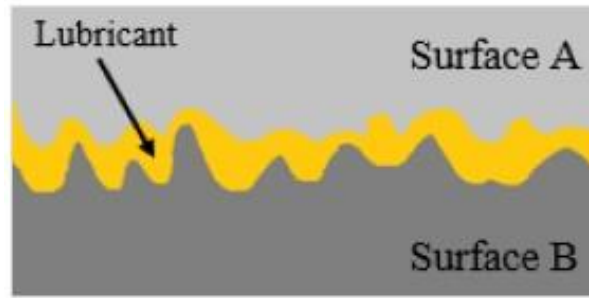


Figure 2.1: Lubrication between two surfaces in contact

## 2.2 Type of lubricants

There are various types of lubricant available in the market nowadays such as liquid, gas, solid and semi-solid lubricant.

### 2.2.1 Liquid lubricant

A liquid lubricant provides special properties such as wear replenishment, low mechanical noise, and the ability to remove wear debris [4]. Liquid lubricants can decompose or oxidise under extremely high temperature and would evaporate under extreme pressure conditions [5]. Liquid lubricants also can be useful as a cooling function and surface protection. Liquid lubricant forms a film in the hydrodynamic and hydrostatic regime. In hydrodynamic regime, the fluid enters the surface by the action of the bearing. Different from hydrodynamic, the fluid is conducted by under pressure from an external source. The bearings are made from hard plastics by rubber materials due to the performance of absorbing vibration, reduce the noise and impact of a system [6]. The lubricant application in the bearing cavity is the main factor to a long lasting surface.

It is also stated that rubber materials with the combination of water is the perfect combination for bearing systems. When the rotation occurs between the shaft and bearing, it is creating a layer of water. This is because of longitudinal grooves that followed by water and radial moves at the middle of the contacting surfaces in a thin film. Under boundary

lubrication conditions, a protective film on the rubbing surface plays an important part on its development as well as managing the wear behaviour [7]. Liquid lubricants are categorised based on the origin, either it was extracted from vegetables, animal fats, mineral oils, or of synthetic origins.

i. **Animal and vegetable oils**

Animal and vegetable lubricating oils are constant oils because they do not evaporate unless they decompose. It is produced by the fatty acids and alcohol compounds in animals. It is usually added to mineral oils to improve film formation.

ii. **Mineral oils**

Mineral oils are the most favourable and economically viable category of liquid lubricant that's been extracted from crude oils. It is stable under service conditions which formed based on carbon and hydrogen, Furthermore, it also contains sulphur, oxygen and nitrogen.

iii. **Synthetic oils**

Synthetic lubricating oils are made up of short and uniform molecules of carbon. This provides the durability to reduce heat and pressure. It is applied when mineral oils are inadequate.

### **2.2.2 Gas Lubricant**

This type of lubricant use gases to lubricate bearings. It is low in viscosity, non-polluting, long lasting and small in frictional loss [4] .Gas lubricants usually performs in aerodynamic or aerostatic regimes at high speed and temperature. Many aircraft industries have utilized this kind of lubricant due to the constant chemical properties over the various ranges of temperature. This lubricant is able to work at high and low

temperatures which will increase the efficiency and reduce cost. Moreover, gas lubricants are reliable due to the appearance of a gas film between the bearing and the shaft to protect from wear [5]. The absence of sealing and contamination problems can also be avoided by the application of gas lubricant.

In fact, gas-lubricated bearing provide the best benefit as it is frictionless, silent, and vibration free. Gas bearings are capable of surviving through extreme conditions for wide surface velocities and are able to reduce the friction coefficient at higher load, sliding speed and sliding distance [4].

### **2.2.3 Solid Lubricant**

Like any other lubricants, solid lubricant provide a thin layer of protection between two surfaces, albeit in powder form instead of liquid. The objective is to develop continuous adherent of soft or hard film in between surfaces which can be applied through mechanical or physical processes. It is applied on one surface before making contact with another.

Solid lubricants are required to reduce the amount of friction and wear under extreme conditions such as critical temperatures, high amounts of dust, and corrosive environment [6]. In addition, solid lubricants are more effective than other lubricants at discontinuous loading, high speeds and high loads in the presence of boundary and mixed regimes. Solid lubricants are generally classified into three different classifications: structural, mechanical and chemical lubricants.

- i. **Structural properties:** comprises layers of lattice-like structures which function as a friction reduction agent. Figure 2.2 shows the presence of layers lattice structures.