

สำนักหอสมุดกลาง พระจอมเกล้าลาดกระบัง

**AN INVESTIGATION ON PHYSICAL AND CHEMICAL
PROPERTIES IN USED LUBRICATING OIL OF DIESEL ENGINE**



E077971

PHIRANAT KHAMSRISUK

เลขหมู่.....**077971**.....
เลขทะเบียน.....**5 ต.ค. 2559**.....
วัน,เดือน,ปี.....



**A THESIS SUBMITTED IN PARTIAL FULFILLMENT
OF THE REQUIREMENT FOR THE DEGREE OF
MASTER OF ENGINEERING IN AUTOMOTIVE ENGINEERING
(INTERNATIONAL PROGRAM)
INTERNATIONAL COLLEGE
KING MONGKUT'S INSTITUTE OF TECHNOLOGY
LADKRABANG
ACADEMIC YEAR 2015
KMITL-2015-IC-M-004-06**

This material is reserved for educational use only, not allowed for commercial use.

Forbidden to modify the content, and cite the document when use.



COPYRIGHT 2015

INTERNATIONAL COLLEGE

KING MONGKUT'S INSTITUTE OF TECHNOLOGY

LADKRABANG

This material is reserved for educational use only, not allowed for commercial use.

Forbidden to modify the content, and cite the document when use.

INDEPENDENT STUDY TITLE AN INVESTIGATION ON PHYSICAL AND
CHEMICAL PROPERTIES IN USED
LUBRICATING OIL OF DIESEL ENGINE
STUDENT NAME MR. PHIRANAT KHAMSRISUK
STUDENT ID 56610009
DEGREE MASTER OF ENGINEERING
PROGRAM AUTOMOTIVE ENGINEERING
ADVISOR ASST.PROF.DR.PREECHAR KARIN
DR.KOBSAK SRIPHAPRA
PROF.DR.KATSUNORI HANAMURA

ABSTRACT

Major functions of lube oil include cooling, friction reduction, and wear control. The lubricating oil develops lubricating film between moving surfaces, which reduces friction and wear. However, the engine oil is a depository of impurities. These are in the form of solid, liquid, and gaseous contaminants. If uncontrolled, these contaminants can build up to excessive levels. High levels of lubricant contamination cause wear of mechanical components as well as breakdown of the lube oil. The result is performance degradation, reduced engine life, and short oil service life. In this research, the study of physical and chemical properties has been done for ten different samples taken from diesel engine. Different aged lubricating oil samples were chosen to see the wearing effect of the engine. The results were compared in terms of wear condition, oil condition, contamination and particles size distribution. Where, wear condition represents the present of total metal particle like iron, aluminum, copper, chromium, oil condition evaluates the properties like viscosity, oxidation, contamination measures presence of soot and difference size of particles.

Keywords: Lubricating oil, Oil filter, Wear condition, Contamination, Laser particles size distribution.

ACKNOWLEDGEMENTS

The study was funded by the Automotive Engineering program under Thailand Advanced Institute of Science and Technology and Tokyo Institute of Technology (TAIST-TokyoTech) which is collaboration between National Science and Technology Development Agency (NSTDA), Thailand, Tokyo Institute of Technology (Tokyo Tech), Japan, and King Mongkut's Institute of technology Ladkrabang (KMITL), Thailand.

I would like to express my special appreciation and gratitude to my advisors Asst. Prof. Dr. Preechar Karin from King Mongkut's Institute of technology Ladkrabang (KMITL), Dr. Kobsak Sriprapha from National Electronics and Computer Technology Center (NECTEC), Thailand, National Science and Technology Development Agency (NSTDA), for their support, technical recommendations, and collaboration all the time throughout the research project. I would like to direct my gratitude to my advisors, Prof. Dr. Eng. H, Tokyo Institute Department of Mechanical and Control Engineering, Graduate School of Science and Engineering, Tokyo Institute of Technology for his recommendations and suggestions.

I would also like to thank Energy Policy & Planning Office, Thailand, for support the scholarship in energy research.

I would also like to thank my committee members, Asst. Prof. Dr. Chinda Charoenphonphanich, and Assoc.Prof.Dr. Jarruwat Charoensuk for their comment and kind support.

I can't thank you enough to Mr. Thanant Sirisithichote, General Manager and Lubrication Specialist of FOCUSLAB Ltd., about great support and very equipment in this research.

Finally, I must express my sincere thanks to my parents and my friends with kindness support and continuous encouragement throughout Master degree student's life. This accomplishment would not have been possible without them. Thank you.

Phiranat Khamsrisuk

CONTENTS

	PAGE
ABSTRACT	i
ACKNOWLEDGEMENTS	ii
CONTENTS	iii
LIST OF TABLES	vii
LIST OF FIGURES	viii
CHAPTER 1	1
INTRODUCTION	1
1.1 Background	1
1.2 Objective of the Study	2
1.3 Scope of the Study	2
1.4 Master Schedule	3
CHAPTER 2	4
LITERATURE REVIEWS	4
2.1 Introduction	4
2.2 Friction and wear	5
2.3 Lubrication	6
2.4 Physical and Chemical properties	7
2.4.1 Viscosity	8
2.4.2 Viscosity index	8
2.4.3 Oxidation	9
2.4.4 Nitration	10
2.4.5 Total acid number (TAN)	11
2.4.6 Total base number (TBN)	11
2.5 Contamination	12
2.5.1 Types of contamination	12

CONTENTS (continued)

	PAGE
2.5.2 Ingression of lubricant contamination	13
2.5.2.1 Built-in contamination	13
2.5.2.2 External ingression	13
2.5.2.3 Internal generation	14
2.5.2.4 Maintenance action	14
2.5.3 Wear and contaminant particle size	15
2.5.4 Soot	16
2.5.5 Fuel contamination	23
2.6 Particle Size Distribution	28
2.7 Biodiesel fuel	29
2.7.1 Biodiesel contamination in lubricating oil	30
CHAPTER 3	32
METHODOLOGY	32
3.1 Sampling	32
3.2 Experimental procedures	35
3.2.1 Experimental procedures of used lubricating oil and oil filter	35
3.2.2 Experimental procedures of fuel contamination	36
3.2.2.1 Equipment of used lubricating oil characteristic test	37
3.2.2.2 Equipment of oil filters test	42
3.2.2.3 Equipment and condition of fuel contamination test	45
CHAPTER 4	48
RESULTS AND DISCUSSIONS	48

CONTENTS (continued)

	PAGE
4.1 Physical properties of used lubricating oil.....	48
4.1.1 Viscosity.....	48
4.1.2 Wear.....	49
4.1.2.1 Wear debris.....	50
4.2 Chemical properties of used lubricating oil.....	55
4.2.1 Oxidation.....	55
4.2.2 Total base number (TBN).....	56
4.3 Contamination in used lubricating oil.....	57
4.3.1 Soot.....	57
4.3.2 Fuel.....	59
4.4 Oil filter.....	60
4.4.1 2D optical microscope.....	62
4.4.2 Scanning electron microscope (SEM) and energy dispersive x-ray spectrometer (EDS).....	63
4.4.3 Particle distribution.....	66
4.5 Biodiesel fuel contamination.....	70
4.5.1 Four ball tester.....	71
4.5.2 3D optical microscope.....	72
4.5.3 Biodiesel fuel model blend with lubricating oil.....	73
4.5.4 Wear scar from SEM image.....	75
4.5.4.1 Abrasive wear.....	76
4.5.4.2 Fatigue wear.....	77
CHAPTER 5.....	78
CONCLUSIONS.....	78
REFERENCES.....	80

CONTENTS (continued)

	PAGE
APPENDICES.....	83
APPENDIX A: Publications.....	84
AUTHOR BIOGRAPHY.....	99



This material is reserved for educational use only, not allowed for commercial use.

Forbidden to modify the content, and cite the document when use.

LIST OF TABLES

	PAGE
Table 1.1 Procedure Plan	3
Table 2.1 Types of diesel engine oil contaminants	13
Table 2.2 Diesel engine component oil film thicknesses	15
Table 2.3 Effect of contaminant size on wear	16
Table 3.1 Engine specification	32
Table 3.2 Lubricating oil SAE 0w-30 specification	33
Table 3.3 Mileage and Oils sample specification	34
Table 3.4 Percent of fuel contamination in four ball test	45
Table 3.5 Condition test of four ball tester	46
Table 4.1 Type and size of wear contaminate in used lubricating oil	54
Table 4.2 Palm biodiesel specification	70

LIST OF FIGURES

	PAGE
Figure 2.1 Four-Ball tribotest schematic.....	18
Figure 2.2 Soot contaminations in heavy duty diesel engine.....	19
Figure 2.3 TEM image at variation of magnification.....	20
Figure 2.4 Carbon black size distributions.....	21
Figure 2.5 Wear on metal balls tested in (a) lubricant A, (b) lubricant A with carbon black.....	22
Figure 2.6 Wear on metal balls tested in (a) lubricant B, (b) lubricant B with carbon black.....	22
Figure 2.7 Linear pin wear as a function of sliding time for various bio-lubricants.....	24
Figure 2.8 Coefficient of friction as a function of sliding time for various bio-lubricants.....	25
Figure 2.9 Effect of 15kg load on frictional torque.....	26
Figure 2.10 Effect of 15kg load on frictional torque.....	27
Figure 2.11 Wear scar of sample oils.....	30
Figure 2.12 Weight loss of cylinder liner.....	30
Figure 3.1 Sample of used lubricating oil compare with new lubricating oil.....	33
Figure 3.2 Schematic diagram of methodology in used lubricating oil and filter.....	35
Figure 3.3 Schematic diagram of methodology in fuel contamination.....	36
Figure 3.4 Fourier Transform-Infrared Spectroscopy (FTIR).....	37
Figure 3.5 Viscosity meter.....	38
Figure 3.6 Wear analysis optical spectrometers.....	39
Figure 3.7 The spectroil analytical ferrography.....	40

LIST OF FIGURES (continued)

	PAGE
Figure 3.8 Type and cause of wear.....	41
Figure 3.9 Scanning electron microscopy (SEM).....	42
Figure 3.10 Schematic diagram of CHNS element analyzers.....	43
Figure 3.11 The laser diffraction granulometer and associated dispersion units.....	44
Figure 3.12 Four ball tester.....	45
Figure 3.13 3D optical microscope.....	47
Figure 4.1 Impact of viscosity at 40°C on mileage and lubricating oil age.....	48
Figure 4.2 Impact of viscosity at 100°C on mileage and lubricating oil age..	49
Figure 4.3 Impact of wear on mileage and lubricating oil age.....	50
Figure 4.4 Total metal wear.....	51
Figure 4.5 Wear element.....	51
Figure 4.6 New oil SAE 0w-30.....	53
Figure 4.7 Wear debris in used lubricating oil.....	53
Figure 4.8 Wear debris in used lubricating oil.....	53
Figure 4.9 Type of wear in used lubricating oil.....	55
Figure 4.10 Impact of oxidation on mileage and lubricating oil age.....	56
Figure 4.11 Impact of oxidation on mileage and lubricating oil age.....	57
Figure 4.12 Impact of soot on mileage and lubricating oil age.....	58
Figure 4.13 Soot distribution in lubricating oil of car number 6.....	59
Figure 4.14 Soot distribution in lubricating oil of car number 9.....	59
Figure 4.15 Impact of fuel contamination on mileage and lubricating oil age..	60
Figure 4.16 Particle before clean in fiber.....	62
Figure 4.17 Particle after clean in fiber.....	62

LIST OF FIGURES (continued)

	PAGE
Figure 4.18 Particle in fiber of oil filter from SEM image.....	63
Figure 4.19 Agglomerate metal particles from EDS test (A).....	63
Figure 4.20 Small metal particles from EDS test (B).....	64
Figure 4.21 Soot particles from EDS test (C).....	64
Figure 4.22 Carbon composite particles from EDS test (D).....	64
Figure 4.23 Element components in each particle.....	65
Figure 4.24 Particle distribution size of used lubricating oil and oil filter all samples.....	67
Figure 4.25 Average particle size in used lubricating oil and oil filters.....	68
Figure 4.26 Size of particle from lubricating system of diesel engine.....	69
Figure 4.27 Impact of biodiesel fuel contamination to friction torque.....	71
Figure 4.28 Impact of biodiesel fuel contamination to wear scar.....	72
Figure 4.29 Deep scars in each sample from 3D microscope.....	72
Figure 4.30 Average deep scars in each sample from 3D microscope.....	73
Figure 4.31 Typical diesel molecules.....	73
Figure 4.32 Typical biodiesel molecules.....	73
Figure 4.33 Model of biodiesel fuel blend with lubricating oil.....	74
Figure 4.34 Wear scar in each blend biodiesel fuel of steel ball from SEM images with magnificent 150 times.....	75
Figure 4.35 Mechanism of abrasive wear.....	76
Figure 4.36 Abrasive wear on ball steel B100-2% from SEM with magnificent 500 times.....	76
Figure 4.37 Mechanism of fatigue wear.....	77
Figure 4.38 Fatigue wear on ball steel B100-2% from SEM with magnificent 1,500 times.....	77

CHAPTER 1

INTRODUCTION

1.1 Background

A major function of lubricating oil is friction reduction and wears control. The lubricating oil develops lubricating film between moving surfaces, which reduces friction and wear. However, the engine oil is a depository of impurities. These are in the form of solid, liquid, and gaseous contaminants. If uncontrolled, these contaminants can build up to excessive levels. High levels of lubricant contamination cause wear of mechanical components as well as breakdown of the lube oil. The result is performance degradation, reduced engine life, and short oil service life. The main cause of lubricating oil declination is contamination such as wear debris, soot particles, oxidation, water, fuel, glycol, air and heat [1].

The way that fuel is injected and ignited, agglomerate soot formation occurs more commonly in diesel than in gasoline engines. Unlike gasoline engines where the fuel/air mixture is ignited with a spark, fuel and air entering the diesel cylinder ignite spontaneously from the high pressure in the combustion chamber. The fuel and air mixture in diesel engines typically do not mix as thoroughly as they do in gasoline engines. This creates fuel-dense pockets that produce soot when ignited. While the majority of soot easily escapes through the exhaust, some gets past the piston rings and ends up in the lubricant oil. These impacts directly on declination life of lubricating oil which contributes to wear [2].

1.2 Objectives

- 1.2.1. To characterize physical and chemical properties of lubricating oil (SAE 0w-30) in used diesel engine.
- 1.2.2. To investigate wear particles size distribution in used lubricating oil (SAE 0w-30) and oil filter in diesel engine by using laser diffraction technique.

1.3 Scope of the study

- 1.3.1 Investigate physical and chemical properties of lubricating oil (SAE 0w-30) from used diesel engine.
- 1.3.2 Investigate the distribution particles size of used lubricating oil and filter of diesel engine by laser diffraction technique.
- 1.3.3 Investigate the wear scar and friction of fuel contamination from synthetic lubricating oil (SAE 0w-30) by compare with blend biodiesel fuel.
- 1.3.4 Investigate the wear particles size distribution of ball wear from four ball test by laser diffraction technique.

1.4 Master Schedule

Table 1.1 Procedure Plan

Plan	2015												2016						
	Jan	Feb	Mar	Apr	May	Jun	Jul	Aug	Oct	Sep	Nov	Dec	Jan	Feb	Mar	Apr	May	Jun	Jul
1. Investigate the physical and chemical of lubricating oil.																			
2. Investigate and studying the data including the journal, literature review.																			
3. Sampling data of used lubricating oil and filter from diesel engine.																			
4. Analyze physical and chemical properties of used lubricating oil and filter.																			
5. Design the experimental procedure of wear condition by using four ball tester.																			
6. Investigate wear particles size distribution from four ball test.																			
7. Analysis.																			
8. Thesis Writing and preparing presentation for publishing and conference.																			
9. Thesis Defense.																			

CHAPTER 2

LITERATURE REVIEWS

2.1 Introduction

A lubricant is a substance used to facilitate relative motion of solid bodies by minimising friction and wear between interacting surfaces. Lubricants can be considered as constructional elements or engineering fluids which have to be kept on the newest technological level because of the continuous development of tribological systems. As a product group, lubricants play an essential role in industry and transportation: without lubrication, there is no transfer of energy. Reduction of friction and wear by use of lubricants enables machinery to work without premature failure, results in energy savings and controls emissions. Related functional fluids are used in applications where their primary purpose is not just friction and wear reduction, although some degree of lubricity may be required. Examples of such applications are hydraulic fluids, electrical transformer fluids, heat transfer fluids and metalworking coolants. Apart from high-volume applications such as engine, gear and hydraulic fluids, there are thousands of other specifically tailored lubricant or grease formulations that differ greatly in composition, both chemically and physically.

Lubricants represent considerable economic and ecological importance. Recently, in this context, saving resources and energy and reducing emissions have become essential matters. Lubricants have a particularly important contribution to make in relation to energy conservation, minimization of waste and development of durable products. Correct lubricant selection can lead to improvements in energy efficiency of up to 10%. Minimising wear by efficient lubrication prolongs the useful life of machines, thereby decreasing the consumption of non-renewable resources. Industrial production processes can reach higher levels of efficiency and products can be made more attractive through the added value offered by lubricants. Truly green lubricants are those that optimise energy efficiency and minimise wear in the machinery they lubricate and have maximised service lifetimes in order to reduce the amount of lubricant required [3]. Adoption of high performance lubricant technologies has led to a significantly lower lubricant consumption.

Modern high-performing commercial lubricants are usually complex materials composed of a lubricant base stock formulated with an additive

package for specific property enhancement of the resulting full lubricant formulation. Traditional lubricant systems are highly diverse, ranging mainly from common lube oils (non-aqueous liquids) to oil-in-water emulsions (e.g. used in water-miscible cutting fluids), water-in-oil emulsions (as in metal-forming), oil-in-oil emulsions (applied in metalworking), water-based solutions (applied in chip-forming metalworking operations), greases and pastes, and solid lubricants. Water is a truly green lubricant or lubricant component. Water-based lubricants possess distinctive advantages over oil based lubricants (such as environmental compatibility, biocompatibility, availability, cost effectiveness). Their environmental compatibility makes them suitable for a number of industrial applications, such as food processing or textile and pharmaceutical manufacturing, where the use of oil-based lubricants can be problematic due to contamination issues. For biomedical applications (such as lubricious coatings for catheters) aqueous lubrication is particularly important, since water is virtually the only acceptable base lubricant.

In this chapter, the reviewed papers will be presented the fundamental of Friction, wear, lubrication, lubricant properties and source wear debris of components in diesel engine, parameters that cause to lubricating oil breakdown and reduce lifetime. Finally, In order to investigate the particle size distribution of wear debris, influenced parameters were considered.

2.2 Friction and wear

Friction is the mechanical force which resists movement (dynamic or kinetic friction) or hinders movement (static friction) between two sliding or rolling surfaces in contact. These types of friction are also known as external friction. The causes of external friction are the microscopic contact points between the sliding surfaces. Depending on the application, friction may be desirable or undesirable. For example, tyre traction and braking rely on the beneficial effects of friction for their effectiveness. On the other hand, in applications such as operation of engines or equipment with bearings and gears, friction is undesirable because it causes wear, generates heat and frequently causes premature failure.

The energy expended in overcoming friction is dispersed as heat and is essentially wasted. Waste heat is a major cause of excessive wear and premature failure of equipment. Friction occurs essentially in two different modes: sliding and rolling friction. The relationships between the frictional force and the load or weight of the sliding object differ for dry, or unlubricated,

surfaces and lubricated surfaces. The coefficient of sliding friction is constant but depends on the type of material [4]. Adhesion occurs at the contacting points of surfaces and refers to the (cold) welding effect that occurs when two bodies are compressed against each other under pressure. A shearing force is required to separate cold welded surfaces. Reduced wear and heat effects may be achieved by inserting a lower-viscosity (shear strength) material between wearing surfaces that have a relatively high coefficient of friction. Any material used to reduce friction in this way is a lubricant.

Much less force is required to roll an object than to slide or drag it. Unlike the coefficient of sliding friction, the coefficient of rolling friction varies with the rolling conditions. Rolling friction is very small compared with sliding friction. Housed ball or roller bearings are examples of practical applications which are subject to the laws of rolling friction. Roller bearings are the most important machine elements. Wear is defined as progressive damage resulting in material loss due to contact between adjacent working parts. Although some wear may be expected during normal operation of equipment, excessive friction causes premature wear, which translates into significant economic loss due to equipment failure and downtime, and cost of spare parts. Friction and wear generate wasted energy that is not renewable. Therefore, wear can be held responsible for an overall loss in system efficiency. The effects of wear are commonly detected by visual inspection of surfaces. Three types of surface damage are: (i) damage in the form of structural changes, plastic deformation or surface cracking, without exchange of material between the contacting parts; (ii) damage with loss of material (wear); and (iii) damage with gain of material (corrosion). Wear can result from abrasion, adhesion, fatigue or corrosion.

2.3 Lubrication

A tribological system consists of four elements: the contacting partners, an interface with an enclosed medium (lubricant), and the environment. Tribosystems are present in all mobile mechanisms, from automotive, industrial and domestic to aerospace applications. In tribosystems, one material slides against another or against itself. For each combination of materials and contact configurations, the tribological variables include the type of movement, normal applied load, sliding distance, speed and frequency, duration of the applied stress, temperature (ambient and contact temperature), atmosphere, moisture, lubricating. Tribometric parameters are friction, wear and temperature. Specific devices, called tribometers, are used to measure friction and wear [5].

Because of its highly interdisciplinary nature, tribology requires efficient cooperation between mechanical engineers, materials scientists, metallurgists, chemists and chemical engineers, physicists, surface scientists, with contributions of specialists in environmental protection and toxicology.

Tribosystems require adequate material selection, wear protection by special coatings and surface engineering, lubrication and lubricants, modern test equipment in laboratories and test fields. Scientific focus on lubrication technology and lubricants is relatively new. Lubrication is critical for wear protection of mechanical systems that should operate for extended periods of time. Lubrication may also be used for other purposes, namely to

- reduce oxidation and prevent rust;
- reduce energy consumption;
- provide insulation in transformer applications;
- transmit mechanical power in hydraulic fluid power applications;
- Seal against dust, dirt, and water (flushing out contaminants).

A major goal of current tribological research is to find effective lubricants for reactive light alloys such as magnesium, aluminium and titanium, which are of interest in the automotive industry in view of their lower densities and the ability to form corrosion-protective surface layers. Molecular interactions studied by nanotribology have impact on emerging uses in microelectro mechanical systems as well as on traditional lubricant applications in the automotive and industrial sector.

2.4 Physical and chemical properties

Effective lubricants, either bio-based or petroleum-based, must meet several performance requirements. They should properly coat load-bearing surfaces to protect against wear and corrosion, reduce friction and not decompose under working conditions. Physical, chemical and film formation properties of base fluids influence the formulated lubricant performance [6]. Under boundary conditions, the friction coefficient is essentially independent of lubricant viscosity, and becomes dependent on the nature of the contacting metal surfaces. Surface additives can be used to modify the surface chemistry

and reduce friction. A lubricant is vital for proper functioning of engines and machines.

2.4.1 Viscosity

The ability to properly coat working surfaces is commonly quantified by measurements of the viscosity and VI. The properties of a lubricant are further determined by certain important characteristics such as compressibility, stability, PP, cloud point (CP), flash point and acid value (AV) [7]. Viscosity and VI are among the most important physical properties of any lubricant. Viscosity mainly controls the hydrodynamic behaviour of lubricants. Viscosity of an oil is essentially a measure of the oil resistance to shear. It is the friction between lubricant molecules or internal resistance to flow. High viscosity implies high resistance to flow. Viscosity varies inversely with temperature, is also affected by pressure and, in some cases, by the shear rate. The viscosity–pressure behaviour is described by the pressure–viscosity coefficient α , the value of which reflects the rate of oil viscosity increase under increasing pressure. Higher pressure enhances the load-carrying capacity of the oil. This property enables use of thin oils to lubricate heavy machinery. Load-carrying capacity also increases with the machine operating speed.

Viscosity is commonly measured as shear stress and shear rate, or as the time required for an oil sample to flow through a standard orifice at a given temperature. Viscosity measured directly by shear, referred to as the dynamic viscosity η , is expressed in centiPoise ($1 \text{ cP} = 1 \text{ mPa s}$). The oil industry usually employs kinematic viscosity ν (dynamic viscosity divided by oil density), expressed in centiStokes ($1 \text{ cSt} = 1 \text{ mm}^2/\text{s}$), conventionally at 40 and 100 °C. The kinematic viscosity values (at 40 °C) are at the basis of the ISO grades for lubricating oils for industrial use. Viscosity as measured in time is expressed in SUS at 100 °F and 210 °F. The effective viscosity of a lubricant in a bearing may be different from the quoted viscosity measured by a standard test method, and the difference depends on the shear rate in the bearing [8].

2.4.2 Viscosity index

VI is an arbitrary, dimensionless number which expresses the resistance of a lubricant to viscosity change with temperature. It is therefore considered a good indicator of the useful temperature range of a lubricant. Before the emergence of synthetic oil, the Dean and Davis viscosity index, defined as a

function of Saybolt Universal viscosities of an oil at 100 °F and 210 °F, was on a scale of 0 to 100 [9]. With the advent of synthetic oils and additives, the scale now ranges from negative values to over 200 (super high VI), but VI still remains as an indicator of quality.

Lubricating oil with high VI displays only small changes in viscosity with temperature, which denotes stable viscosity. The higher its VI value, the greater the resistance of a lubricant to thicken at low temperatures and thin out at high temperatures. The VI of an oil is crucial in selecting a lubricant for a given application. Lubricants with $VI > 100$ are often considered useful in many applications. Failure to use an oil with the proper VI when temperature extremes are expected may result in poor lubrication and equipment failure. Knowledge of VI is particularly relevant in extreme climatic conditions. An ideal lubricant for most purposes would possess the same viscosity at all temperatures. All lubricants depart from this ideal, some more than others. For example, lubricating oils derived from highly paraffinic crudes have higher VI values than lubricating oils derived from highly naphthenic crudes. This difference was used, in fact, to fix the limits of 0 to 100 on the Dean and Davis scale, these values having been assigned to poor naphthenic base oil and good paraffin-base oil, respectively.

Molecules having a high VI typically consist of long alkyl chains, have low aromatic content, no ethyl branching and no tertiary carbons. Bio-oils have a better VI than mineral oil products, without the need to add shear sensitive additives. The higher density of bio-oils also determines a higher dynamic to kinematic viscosity ratio. Dynamic viscosity is important for lubrication.

2.4.3 Oxidation

Oxidation of lubricating oils depends on the temperature, amount of oxygen contacting the oil, and the catalytic effects of metals. If the oil's service conditions are known, these three variables can be adjusted to provide a test that closely represents actual service. However, oxidation in service is often an extremely slow process, so the test may be time consuming. To shorten the test time, the test temperature is usually raised and catalysts added to accelerate the oxidation. Unfortunately, these measures tend to make the test a less reliable indication of expected field performance. As a result, very few oxidation tests have received wide acceptance, although a considerable number are used by specific laboratories that have developed satisfactory correlations for them. One

oxidation test that is widely used is ASTM D 943, Oxidation Characteristics of Inhibited Steam-Turbine Oils.

This test is commonly known as TOST (turbine oil stability test). While TOST is intended mainly for use on inhibited steam turbine oils, it has been used for hydraulic and circulation oils, and for base oils for use in the manufacture of turbine, hydraulic, and circulation oils. The test is operated at a moderate temperature (95°C, 203°F). Iron and copper catalyst wires are immersed in the oil sample, to which water is added. Oxygen is bubbled through the sample at a prescribed rate [10].

The test is run either for a prescribed number of hours, after which the neutralization number of the oil is determined, or until the neutralization number reaches a value of 2.0. The result in the latter case is then reported as the hours to a neutralization number of 2.0. Objections to ASTM D 943 are that extremely long test times, often on the order of several thousand hours, are required for stable oils, and that the only criterion is the neutralization number.

Severe sludging and deposits on the catalyst wires can occur with some oils without excessive increase in the neutralization number. A modification of the procedure, called procedure B, overcomes some of these latter objections by requiring a determination of the sludge content [11].

2.4.4 Nitration

Nitration: The combustion chambers of engines provide one of the few environments where there is sufficient heat and pressure to break the atmospheric nitrogen molecule down to two atoms that can react with oxygen to form nitrous oxides (NO_x). This becomes a major problem for some engines, especially EGR engines. When nitrogen oxide products enter the lube oil through EGR and normal blow-by, they react with moisture present in the lube and become very acidic and rapidly accelerate the oxidation rate of the oil.

The GCF Filter controls the effects of nitration in the same ways it controls oxidation. By delivering cleaner oil to offer as a seal between the ring and liner, blow-by of NO_x is kept to a minimum. Also, the GCF Filter keeps the oil chemically dry and prevents the mixing of NO_x and moisture, which controls NO_x acid formation and accelerated oxidation of the oil [12].

2.4.5 Total acid number (TAN)

The total acid number (TAN) of oil is synonymous with neutralization number. The TAN of oil is the weight in milligrams of potassium hydroxide required to neutralize one gram of oil and is a measure of all the materials in oil that will react with potassium hydroxide under specified test conditions. The usual major components of such materials are organic acids, soaps of heavy metals, intermediate and advanced oxidation products, organic nitrates, nitro compounds, and other compounds that may be present as additives. It is worth mentioning that new and used oil can exhibit both TAN and TBN (total base number) values.

Organic acids may form as a result of progressive oxidation of the oil, and the heavy metal soaps result from reaction of these acids with metals. Mineral acids (i.e., strong inorganic acids), if present in an oil sample, are neutralized by potassium hydroxide and would, therefore, affect the TAN determination. However, such acids are seldom present except in internal combustion engines using high sulfur fuels or in cases of contamination [13].

2.4.6 Total base number (TBN)

The total base number of oil is the quantity of acid, expressed in terms of the equivalent number of milligrams of potassium hydroxide, which is required to neutralize all basic constituents present in one gram of oil. This test is normally used with oils that contain alkaline, acid-neutralizing, additives. The rate of consumption of these alkaline materials (TBN depletion) is an indication of the projected serviceable life of the oil. With used oils, it indicates how much acid-neutralizing additive remains in the oil. Typical oils of this nature include diesel engine oils for internal combustion engines that use fuels containing acid-producing constituents such as sulfur or chlorine. As long as any significant amount of TBN remains in the oil, there should not be any strong acids present. However, the nature of high alkaline and metallic antioxidant additives sometimes allow for both TAN and TBN values to be obtained on the same sample. This occurs for both new and used oils [14].

2.5 Contamination

The diesel engine is receiving increasing attention as a power plant because of its fuel efficiency and life advantages. Over seventy percent of the trucks manufactured today use diesel engines. Major functions of lube oil include cooling, friction reduction, and wear control. The lube oil develops a lubricating film between moving surfaces, which reduces friction and wear. However, the engine oil is a depository of impurities. These are in the form of solid, liquid, and gaseous contaminants. If uncontrolled, these contaminants can build up to excessive levels.

High levels of lubricant contamination cause wear of mechanical components as well as break-down of the lube oil. The result is performance degradation, reduced engine life, and short oil service life [15].

2.5.1 Types of contamination

Lubricant contaminants degrade the life and performance of diesel engines. These contaminants fall into three categories

- Solid particles, including wear debris, which damage mechanical components and catalyze lubricant breakdown.
- Liquid contaminants, including fuel and water, which hinder the proper operation of the lubricant and its additives.
- Gaseous contaminants, including combustion products, which corrode component surfaces and break down the oil.

The predominant types of diesel engine oil contaminants, along with primary sources and major problems these impurities cause, are listed in Table 2.1

Type	Primary Sources	Major Problems
Metallic Particles	Engine Wear	Abrasion, Fatigue and Lubricant Breakdown
Metal Oxides	Engine Wear and Corrosion	Abrasion and Fatigue Corrosion
Sand and Dust	Combustion Blowby	Abrasion and Fatigue

soot	Combustion Blowby	Lubricant Breakdown
Exhaust Gases	Combustion Blowby	Lubricant Breakdown
Fuel	Combustion Blowby	Lubricant Breakdown
Water	Combustion Blowby	Corrosion and Lubricant Breakdown
Acids	Combustion Blowby and Lubricant Breakdown	Corrosion

Table 2.1 Types of diesel engine oil contaminants, along with primary sources and major problems

2.5.2 Ingression of lubricant contamination

Contamination enters the engine lube system by four routes: 1) built-in from manufacture and assembly, 2) external ingression, 3) internal generation, and 4) maintenance actions.

2.5.2.1 Built-in contamination

Diesel engine manufacturers take great care during the manufacture and assembly processes to ensure high quality control. However, casting materials, machining swarf, abrasives, polishing compounds, and even lint remain after manufacture and overhaul. These built-in contaminants can rapidly damage moving engine parts

2.5.2.2 External ingression

External ingression is a major source of hard particle contamination. Airborne particles, in the form of sand, salt, and other minerals, enter through the engine intake system and mix with the atomized fuel, which is compressed and then burned. Since most of these particles have melting points considerably above the temperatures reached in the diesel combustion process, they remain hard abrasive solids. Airborne contamination has been shown to be the greatest cause of ring-to-cylinder wear. The strong pressure shock wave created during

combustion forces gases through the piston ring clearances. This process, known as blowby, carries particles into the engine oil. Particles may also be retained in the oil film. They are then wiped by the rings into the oil sump on the next down stroke of the piston.

Exhaust gases are similarly driven into the lube oil as blowby gases. These exhaust gases include unburned fuel, water, nitrous oxide, soot, and other partially burned hydrocarbons. Higher levels of exhaust gas recirculation have been shown to increase lube oil particulate contamination. New rules issued by the Environmental Protection Agency requiring stricter nitrous oxide and exhaust particulate controls take effect in 1988. Although new ceramic combustion technologies are under development to reduce exhaust gas particles, it is likely that these regulations will lead to higher percentages of exhaust gas recirculation, resulting in further deterioration of oil properties [16].

2.5.2.3 Internal generation

Internal generation of contaminants is by wear of mechanical component parts and by lubricant breakdown. Mechanical component wear from abrasion, fatigue, adhesion, and corrosion releases harmful particles into the oil. The wear debris is in the form of hard metal particles and of abrasive metal oxides. Wear debris particles of sizes not controlled by standard filtration can build up to grossly contaminate the lube oil.

Lubricant breakdown is the loss of important properties of the oil plus accumulation of harmful materials derived from the oil. These materials include acids, sludges, gels, and additive precipitates. These contaminants can wear moving component parts as well as clog flow passages and heat exchange surfaces.

If wear debris and materials from lubricant breakdown accumulate in the oil, the result is more wear, generating more contaminants. The process of particles wearing surfaces and generating new particles that in turn cause more wear is known as the chain-reaction-of-wear.

2.5.2.4 Maintenance action

During maintenance activities contaminants are introduced into the lubrication system. Opening rocker covers, the engine head, even the oil filler cap allows entrance of dust and water. Simply making and breaking a fitting

generates tens of thousands of damaging particles. In addition, new oil contains contaminant particles.

2.5.3 Wear and contaminant particle size

There is an important relationship between the size of contaminant particles and the thickness of dynamic lubricant films separating opposing surfaces. Particles the size of and larger than the lubricant film thickness cause wear of component surfaces. These particles bridge the gap maintained by the oil film, making simultaneous contact with both surfaces. This focuses the force between the surfaces, causing damage and resulting in component wear. An extensive survey of the technical literature for oil film thicknesses in diesel engine components is summarized in Table 2.2

Component	Oil Film Thickness
Ring/Cylinder	0.3-7
Rod Bearings	0.5-20
Main Shaft Bearings	0.8-50
Turbocharger Bearings Piston	0.5-20
Pin Bushing	0.5-15
Valve	0-10
Train Gearing	0-1.5

Table 2.2 Diesel engine component oil film thicknesses

Most of these dynamic clearances are between 0 and 20 microns. Contaminant particles the size of or larger than these dynamic clearances produce a major portion of the wear experienced by diesel engine oil wetted components. As a demonstration of the link between contaminant particle size and oil film thickness, the results of a diesel engine wear are summarized in Table 2.3 In this test contaminant particles in different size ranges were evaluated for effect on engine wear. It was demonstrated that particles the size of the dynamic oil clearances produced the most diesel engine wear. Even contaminants containing large particles up to 80 microns did not cause as much damage as did contaminants with particles concentrated in the 0 to 10 micron size range.

Dust Size (μm)	Wear Rate(mg/hr)			Wear	
	Mains	Rod	Rings	Total	Relative
0-5	11.9	14.4	28.4	54.7	3.3
5-10	16.1	20.0	23.6	59.7	3.64
0-80	6.2	5.1	14.5	28.8	1.76
10-20	7.4	4.6	4.4	16.4	1.00

Table 2.3 Effect of contaminant size on wear

2.5.4 Soot

Soot production in diesel engine can happen because the internal combustion engines produce soot as a result of incomplete fuel combustion. Ideally, complete combustion in a cylinder would only produce carbon dioxide and water, but no engine is completely efficient.

soot occur more in diesel engines Because of the way that fuel is injected and ignited, soot formation occurs more commonly in diesel than in gasoline engines. Unlike gasoline engines where the fuel/air mixture is ignited with a spark, fuel and air entering the diesel cylinder ignite spontaneously from the high pressure in the combustion chamber. The fuel and air mixture in diesel engines typically do not mix as thoroughly as they do in gasoline engines. This creates fuel-dense pockets that produce soot when ignited. While the majority of soot easily escapes through the exhaust, some gets past the piston rings and ends up in the oil. Factors that Cause Excessive Soot Levels in Crankcase Oil:

- Periods of excessive idling
- Worn piston rings
- Injectors with poor fuel spray patterns
- Rich air-fuel ratios
- Clogged air filters decreasing the air supply, which increases the fuel-air ratio and ultimately leads to increased soot formation

Soot particles are 98 percent carbon by weight and typically spherical in shape. While most are only around .03 microns in size, they often clump together to form larger particles. Individual soot particles pose little risk to engine parts, but clumps of soot can cause damage. Dispersant additives in today's engine oils keep the individual soot particles from forming damaging clumps.

From the literature review Chokun Supanamok and his team investigated "Impact of Soot in Engine Lubricating Oil on Metal Wear using Four-Ball Testing" Soot particle contamination was simulated using carbon black (CB). The candidate lubricants were two formulated engine lubricants for heavy duty diesel engine which have the viscosity grade as SAE 15W-40 and also blended with carbon black. Effects of oils with different additive on size distribution, morphology, and nanostructure of particles were studied [17].

Transmission Electron Microscopy (TEM) was employed to study the morphology and nanostructure parameters of carbon black particles and agglomerated carbon black. The tribometer ball wear surfaces were analyzed by Optical Microscopy (OM).

The Four-Ball testing is one of the tribological test methods. This test method can be used to determine the relative wear preventive properties of lubricating fluid in sliding contact under the prescribed test conditions. No attempt has been made to correlate this test with balls in rolling contact As Figure 2.1 the Four-Ball tribotest is a versatile test for evaluating seizure and wear. The upper holder has one rotating steel ball, which is loaded against three stationary lower steel balls.

All contact areas are drowned in the test lubricant. The load can be applied by using deadweight or through a hydraulic system. The rotation is central along the symmetry axis of both the upper and the lower holders. Circular wear scars will appear on the lower balls, while a circular wear track will appear on the upper ball.

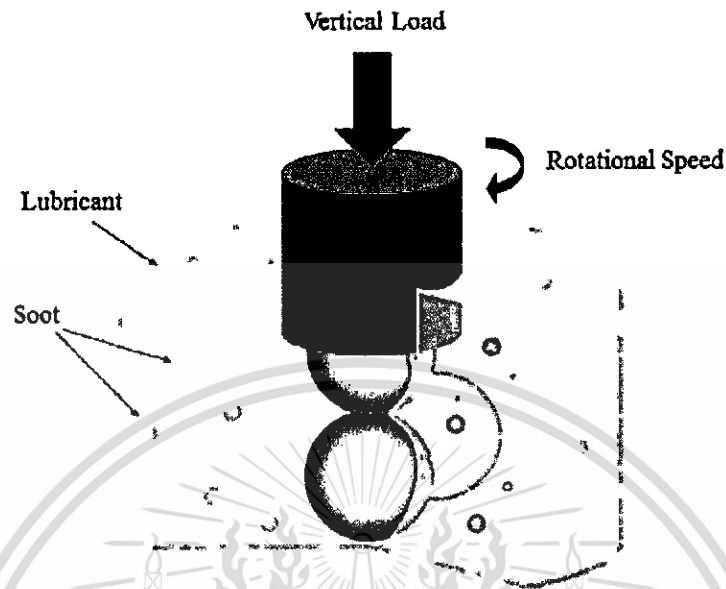


Figure 2.1 Four-Ball tribotest schematic

Three 12.7 mm diameter steel balls are clamped together and covered with the lubricating oil sample to be evaluated. A fourth 12.7 mm diameter steel ball, referred to as the top ball, is pressed with a force of 392 N into the cavity formed by the three clamped balls for three-point contact. The temperature of the test sample is regulated at 75°C and then the top ball is rotated at 1200 rpm for 60 min.

Lubricating oil samples are compared by using the average size of the scar diameters worn on the three lower clamped balls and friction torque is also measured. The wear scar results are observed by Optical Microscopy (OM).

After testing lubricant A on heavy duty diesel engine truck long lasting 30,000 km, the results have been shown on the graph in Figure 2.2 The graph showed the amount of soot that concentrate in lubricating oil over the period of route trip. For lubricant A, it grew up from 0.43 at first sampling (at 8,000 km) to 0.47 %wt at the end of test (about 30,000 km).

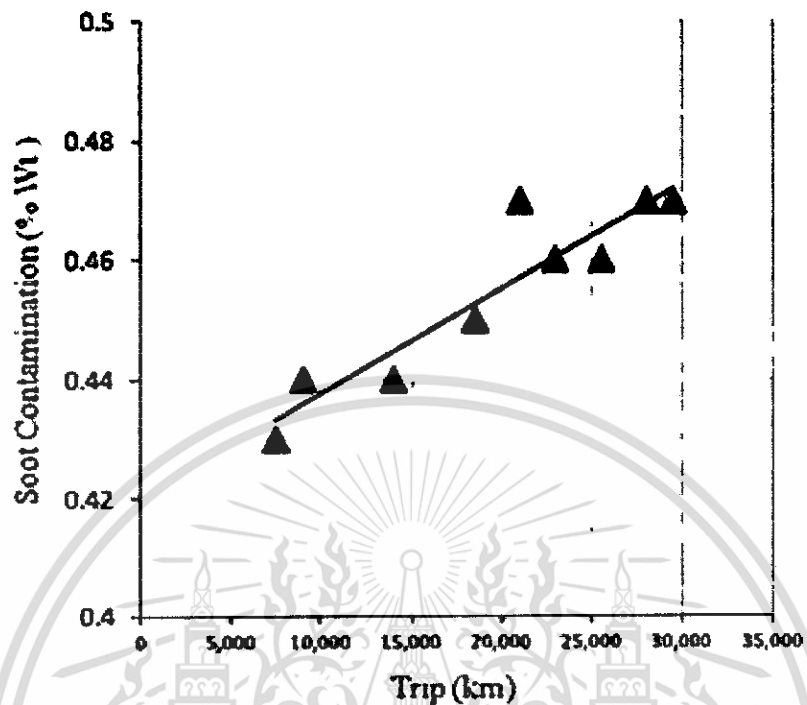


Figure 2.2 Soot contaminations in heavy duty diesel engine

The images shown in Figure 2.3 illustrate typical soot agglomerates from commercial carbon black sample prepared using the ethanol solvent extraction technique and put it on copper grid plate. The agglomerates have a branching structure and typically fall in the size range 30-700 nm. Figure 2.3 consisted of 4 types of image which vary by the magnification of microscope. The most rough one was captured the view of large soot or carbon black agglomerates with a scale at 500 nm.

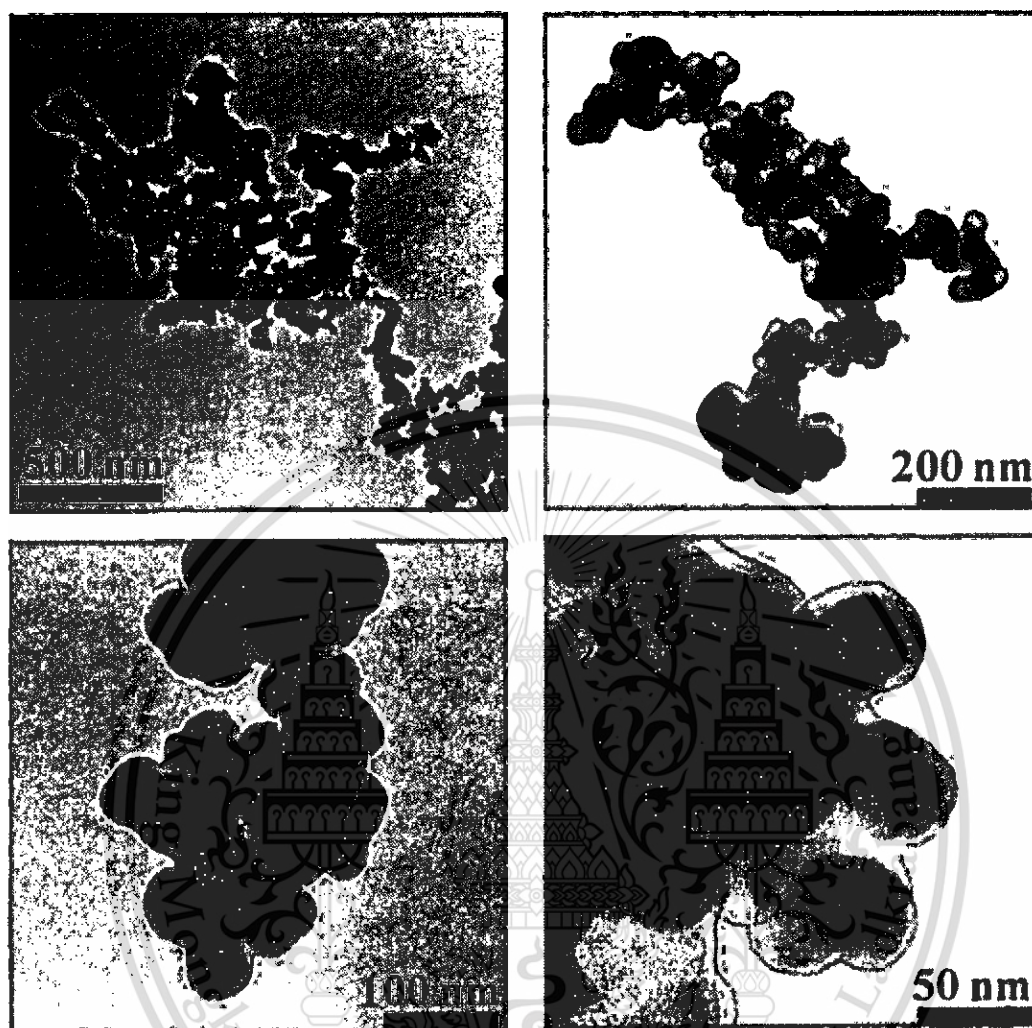


Figure 2.3 TEM image at variation of magnification

286 primary carbon black particulates were measured and the result of all measuring has been plot on bar chart as a histogram graph in Figure 2.4. An average size is 58 nm. The minimum size and maximum size are 30 nm and 87 nm respectively with standard deviation of 11 nm.

The highest peak on histogram graph is at the rage of 51 - 55 nm in size. As of dividing size range with standard deviation value, the major particles are fallen in the size range 46 -70 nm which is accounted for 74% of 286 particulate samples. Moreover, a number of smaller size and bigger size than major one are 14% and 12% respectively. This result differed from all reviewed literatures in the introduction section. They all got the primary size of soot near to 30 nm

which contradict to this result showing that major size is near to 58 nm. It could be an effect from carbon black production process.

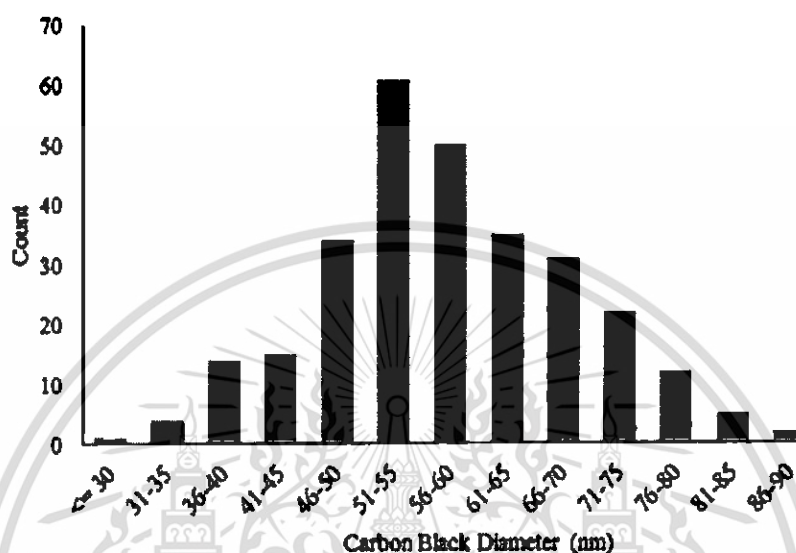


Figure 2.4 Carbon black size distributions

Figure 2.5 shows the microscopy image of wear scars found on three lower balls after 60 min running time in lubricant A without carbon black (on left hand side) and lubricant A with carbon black (on right hand side). The results showed that wear diameter of three metal balls in case of testing

With soot are larger than wear diameter of three balls without soot. Similarly, wear diameter of three balls in lubricant B with soot are larger than wear diameter of three balls without soot as shown in Fig. 6. Wear edge shape of balls in lubricant A are quite smoother than shape of balls in lubricant B. The average wear diameter of balls in case of testing in lubricant A is 786.3 microns and in lubricant A with soot is 877.3 microns. In case of lubricant B, the average of wear diameters are 723.5 and 790.6 microns for case without soot and with soot respectively. Even though they are same viscosity grade, wear shapes are a little bit different.

The conclusion carbon black was contaminated in two conventional SAE 15W-40 engine lubricating oils and tested in Four-Ball Tribotest for evaluate wear behavior. The results showed that carbon black could lead to result in increased wear diameter and made wider wear scar than pure SAE 15w-40.

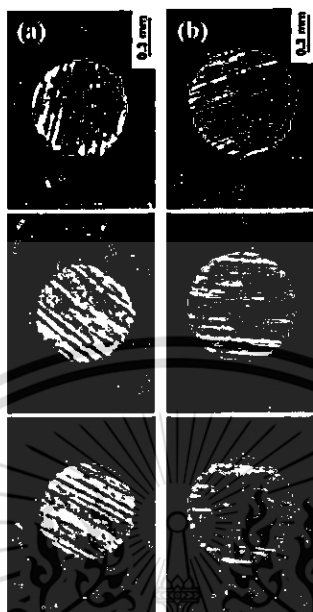


Figure 2.5 Wear on metal balls tested in (a) lubricant A, (b) lubricant A with carbon black

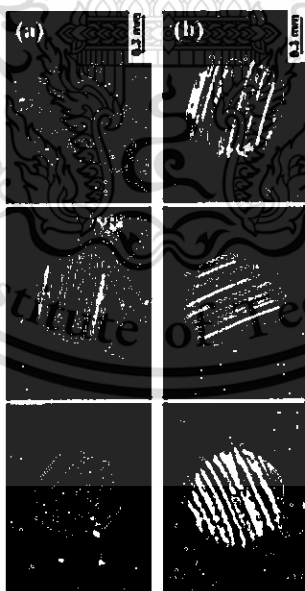


Figure 2.6 Wear on metal balls tested in (a) lubricant B, (b) lubricant B with carbon black

2.5.5 Fuel contamination

The revolution in industrialization throughout the world has boosted the utilization of machinery and shifted the muscle power to machines. The use of machines in various sectors has increased the demand of petroleum based lubricants. Lubricants act as an antifriction media, facilitating smoother working, reducing the risks of undesirable frequent failures and maintaining reliable machine operations among different rotating parts of machines. Due to the depleting of petroleum resources and environment concern, the demand of non-edible vegetable oil based lubricants has increased as well.

A.Imran and his team investigated “Friction and Wear Characteristic of Jatropha Oil Blended Lube Oil” The experiment has been conducted on aluminium pins and cast iron disc which lubricated with Jatropha oil blended bio-lubricant (JBL). To prepare the bio-lubricant, 0%, 20%, 30%, 40% and 50% by volume of Jatropha oil were blended with lubricant SAE 40. In order to understand the characteristics of lubricant, viscometer and multi oil analyser tests have been conducted. It has been found that 10% of Jatropha oil bio-lubricant gives lowest wear and creates less amounts of heat than others samples, and with above 10% contamination, the wear and lubricating temperature increases significantly [18].

Fig 2.7 show the curves of pins wear as a function of sliding time for various Jatropha oil blended with lubricant SAE 40. The values of linear pin wear under 2000 rpm and 30 N loads for each pin vary from 0.02 to 0.05 mm. It was observed that the higher or maximum wear occurred in the beginning of the experiment for some of the test specimens. It is clear from graph that maximum wear occurred for JBL40 and for JBL10 wear is minimum.

We also can observe from the graphs that except JBL40, for each JBL, pin wear decreases gradually and constantly. In the beginning of the test, we can see the wear rate was fast in the period of time that is called the running-in period. During this section, the asperities of the sliding surface are cut off and the contact area of the sliding surface grows to an equilibrium size. We can see from our daily life, that is why we need to service new cars after short period of time running

The graphs of different JBL curve are difference and decrease almost steady throughout this state. It means that different JBL give difference rate of wear to the specimens. Generally, the graphs may be divided into two groups which is the first group is higher value of pin wear and the second group is lower value of pins wear. It can be seen that JBL30, JBL 40 and JBL50 have high value of wear while pure lubricant SAE 40, JBL10 and JBL20 have low value of pin wear and their value are nearly with each other.

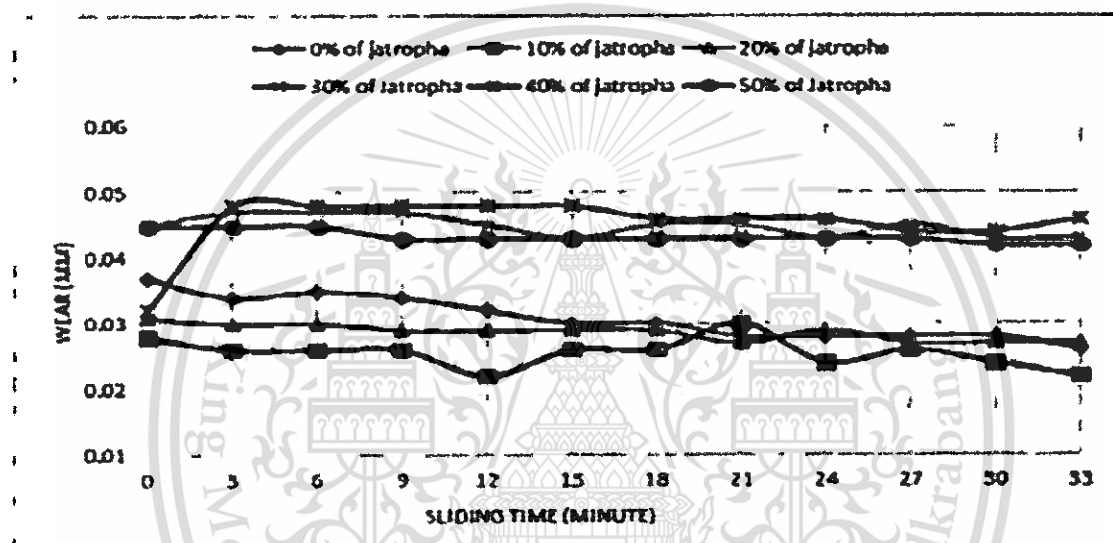


Figure 2.7 Linear pin wear as a function of sliding time for various bio-lubricants.

Fig.2.8 shows the curves of friction coefficient plotted against the sliding time for various Jatropha oil based biolubricants. The results in fig. 4 depict that the lubricant regimes that occurred during the experiment were the boundary lubrication where the value of friction coefficient (μ) for boundary lubricant is in the range of 0.001 to 0.2 except for JBL50. For 0% of Jatropha oil based biolubricants, it can be seen that the coefficient of friction is highest at the beginning and then it falls down rapidly up to minimum value compared to all samples. This phenomenon can be explained by attributing oxide layer on the aluminium surface.

These Lubricant additives react with oxide layer which causes to form a thick tribo-film on aluminium surface. At the initial stage, the shear stress of the tribo-film is high, thus, coefficient of friction is high at early time. With continuing the sliding, the aluminium pin is eroded and fresh metal surface is exposed. Since fresh metal surface has lower tendency to react with lubricant additives, the formation rate of tribo-film decreases than beginning. Therefore, shear stress becomes lower than beginning and lower friction force is experienced. As a result C.O.F decreases with increasing the sliding time.

The difference between the values of coefficient of friction is of Jatropha oil based bio-lubricants and pure lubricant (except for JBL50) is very small which ensures the apt of Jatropha oil based bio-lubricants as lubricant. The JBL10 and JBL30 shows the same C.O.F which is almost 0.15 and JBL50 shows almost same C.O.F throughout the whole operation time which is 0.235.

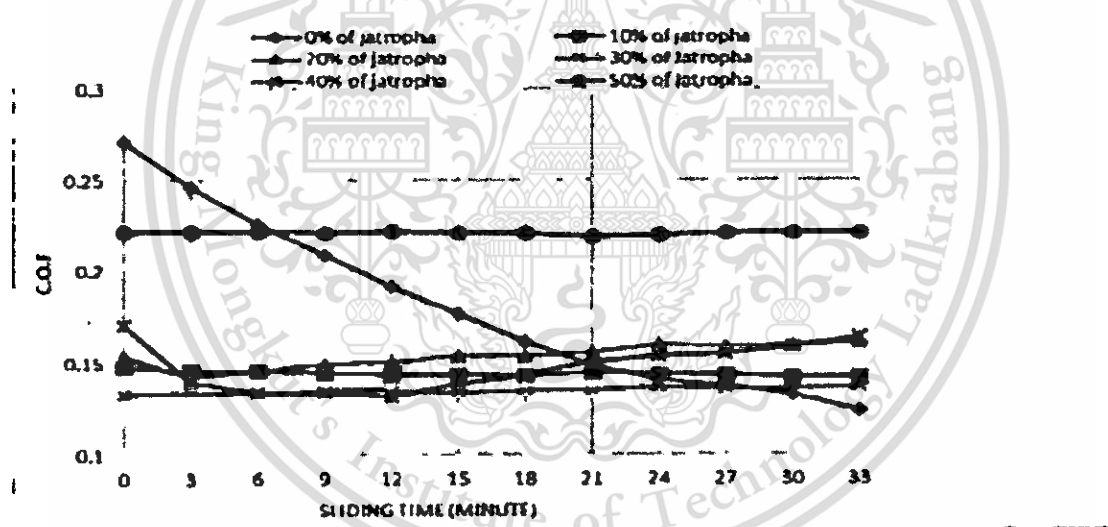


Figure 2.8 Coefficient of friction as a function of sliding time for various bio-lubricants

The rate of wear for various percentage of Jatropha oil based bio-lubricant was different. However, the rate of wear for 10% and 20% of Jatropha oil based bio-lubricant are near to the pure lubricant SAE 40. In this experiment, temperature of lubricating oil increases with sliding increasing time for each percentage of Jatropha oil based bio-lubricant. However, JBL10 showed significant performance as it generates less amount of heat compared to

the other samples. In this experiment, it has been found that having lower wear resistance bio-lubricant contains higher coefficient of friction.

M. Habibullah and his team investigate the effect of Jatropha oil doped with lube oil on tribological characteristics of IP 239 standard using four ball tribotester. The ball test material was EN31 steel, 12.7mm in diameter with a surface finish of 0.1 micrometer center line average (CLA). The test was performed at temperature of 750c with different loads (15 kg and 40 kg) where rotating speed was 1500 rpm and test duration of 3600s. The lube oil used for this experiment was SAE 40 and contaminated with a various blends like 1%, 2%, 3%, 4%, 5%, of jatropha oil. The lubricants were characterized by viscosity using viscometer [19].

Friction torque is the torque caused by the frictional force that occurs when two objects in contact move. Fig. 2.9 and 2.10 shows the variation of friction torque for different percentages of Jatropha oil at various load (15kg and 40kg). Fig 2.9 depicts that J1 exerts highest friction torque while the J5 is lowest at 15 kg load. It is also clear from Fig. 2.10 that the J1 bio-lubricant shows maximum friction torque with compared to other bio-lubricants at 40 kg load. The results from the graph can be attributed to the fact that the bio-lubricants especially, J0 and J1 has strong friction reducing additives as well as ability to retain its property.

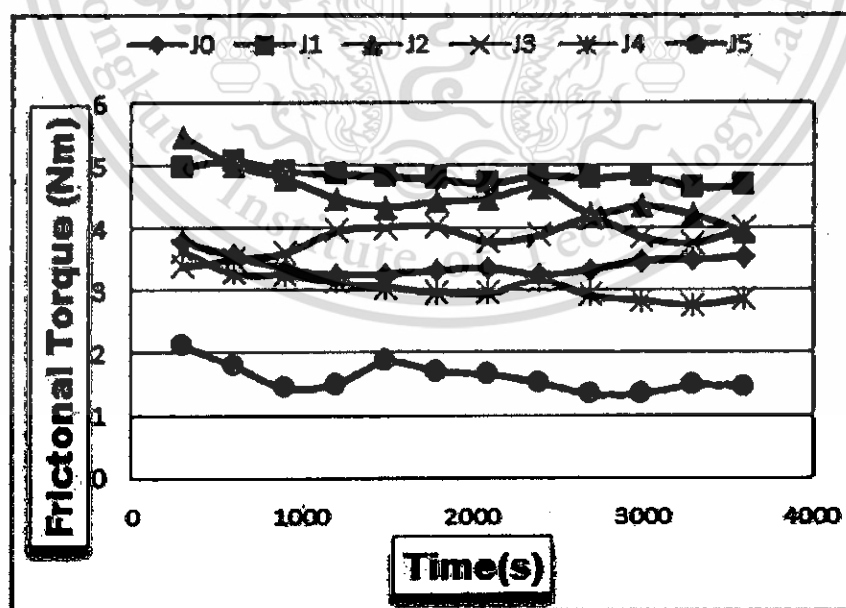


Figure 2.9 Effect of 15kg load on frictional torque.

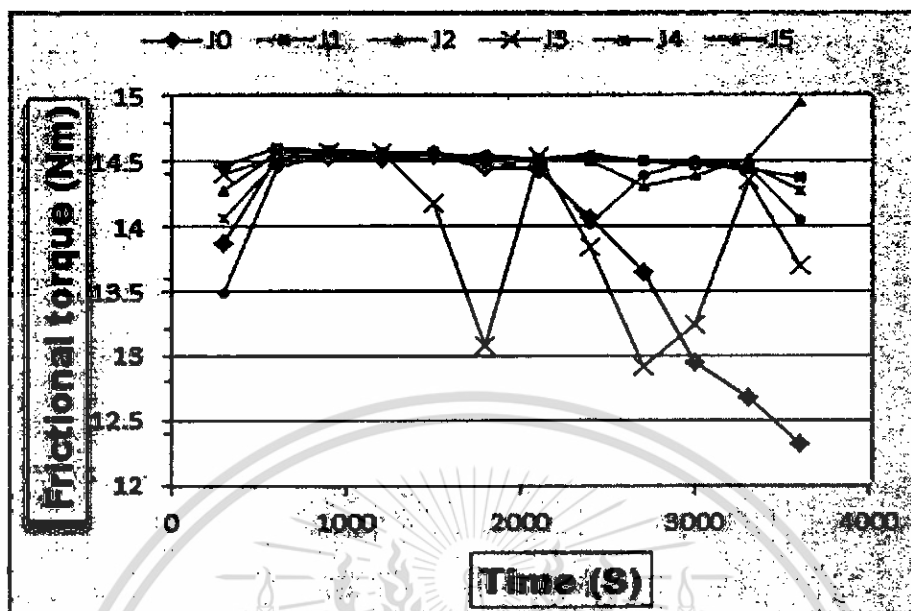


Figure 2.10 Effect of 15kg load on frictional torque.

From the experiment the wear scar diameter (WSD) is more for 40 kg load compared to 15 kg load. That means, at lower loads the WSD under Jatropa contaminated lubricant (SAE 40) are lower, where at higher loads, the WSD are higher and the results shows that the contaminations of lube oil at 1% to 2% of Jatropa oil at 15kg load give higher WSD. While for 40kg load tested, maximum value of WSD stated at 1% and 3% of Jatropa oil. But then, at 5% of Jatropa oil, the value of WSD stated that it was the lowest. Which means 5% of Jatropa oil got the lesser scars and made it the best anti-wear.

The higher value of flash temperature parameter (FTP) clearly observed when 5% of Jatropa oil was used. The 5% of Jatropa oil improves the lubricant (SAE 40) performance, indicating less possibility of lubricant film breakdown. From the observations on worn surfaces of these specimens, 5% of Jatropa oil contaminated lube oil shows better anti-wear lubricant properties than others. For the contamination of Jatropa oil with lube oil, the highest value of kinematic viscosity was stated for 5% at both two temperatures tested (40oc and 100°c).

2.6 Particle Size Distribution

From the literatures, we have been found several size of contaminate particle such as particle of soot, wear element, sand, dust and etc. Lihui Dong et al. Effect of lubricating oil on the particle size distribution and total number concentration in a diesel engine and the result found the peak of particle size number distribution appears at around 75 nm to 100 nm when lubricated with CI oil, while appears at around 87 nm to 115 nm when lubricated with CF oil. The concentration of the highest peak when lubricated with CF is higher than that with CI oil [20].

James A. Addison [16] investigated contamination of diesel lubricant. In this test contaminant particles in different size ranges were evaluated for effect on engine wear. It was demonstrated that particles the size of the dynamic oil clearances produced the most diesel engine wear. There was various particles size that appears in the experiment since 5-100 μm by wear between piston-ring and cylinder range up to 30 μm in size and are work hardened.

Mridul Gautam et al [21]. Studied Effect of diesel soot contaminated oil on engine wear investigation of novel oil formulations the result showed 0.2–0.5% of the fuel mass is emitted as small particles (approximately 0.1 μm in diameter) which consist primarily of soot. Zhu et al [22]. He found that the range of soot particle is 20-35 nm with a nearly normal distribution.

M.A. Fazal et al [23]. investigated friction and wear characteristics of palm biodiesel. The result sizes of the particles removed from cavities of the worn balls in B0-B50 are much bigger than 20 μm cause by adhesive wear.

Hengzhou Wo et al [24]. investigated Morphology, composition, and structure of carbon deposits from diesel and biodiesel oil/diesel blends on a pimple type fuel injector nozzle the result found the carbonaceous deposition of from the emulsified biomass oil/diesel blended fuel was greater than that of diesel. The diameter of deposited carbon agglomerated particulates was approximately 10–30 μm with diesel and 50 μm with the mulsified biomass oil/diesel blend.

2.7 Biodiesel fuel

An alternative diesel fuel that is steadily gaining attention and significance is biodiesel, which is defined as the monoalkyl esters of vegetable oils and animal fats. Low blend levels of biodiesel can restore lubricity to (ultra-)low-sulfur petroleum-derived diesel (petrodiesel) fuels, which have poor lubricity. This feature has been discussed as a major technical advantage of biodiesel. The lubricity of numerous fatty compounds was studied and compared to that of hydrocarbon compounds found in petrodiesel. The effects of blending compounds found in biodiesel on petrodiesel lubricity were also studied.

Lubricity was determined using the high-frequency reciprocating rig (HFRR) test. Dibenzothiophene, which is contained in nondesulfurized petrodiesel, does not enhance petrodiesel lubricity. Fatty compounds possess better lubricity than hydrocarbons, because of their polarity-imparting O atoms. Neat free fatty acids, monoacylglycerols, and glycerol possess better lubricity than neat esters, because of their free OH groups. Lubricity improves somewhat with the chain length and the presence of double bonds. An order of oxygenated moieties enhancing lubricity ($\text{COOH} > \text{CHO} > \text{OH} > \text{COOCH}_3 > \text{C=O} > \text{C-O-C}$) was obtained from studying various oxygenated C10 compounds. Results on neat C3 compounds with OH, NH₂, and SH groups show that oxygen enhances lubricity more than nitrogen and sulfur.

Adding commercial biodiesel improves lubricity of low-sulfur petrodiesel more than neat fatty esters, indicating that other biodiesel components cause lubricity enhancement at low biodiesel blend levels. Adding glycerol to a neat ester and then adding this mixture at low blend levels to low-lubricity petrodiesel did not improve petrodiesel lubricity. However, adding polar compounds such as free fatty acids or monoacylglycerols improves the lubricity of low-level blends of esters in low-lubricity petrodiesel. Thus, some species (free fatty acids, monoacylglycerols) considered contaminants resulting from biodiesel production are responsible for the lubricity of low-level blends of biodiesel in (ultra-)low-sulfur petrodiesel. Commercial biodiesel is required at a level of 1%-2% in low-lubricity petrodiesel, which exceeds the typical additive level, to attain the lubricity-imparting additive level of biodiesel contaminants in petrodiesel[25].

2.7.1 Biodiesel contamination in lubricating oil

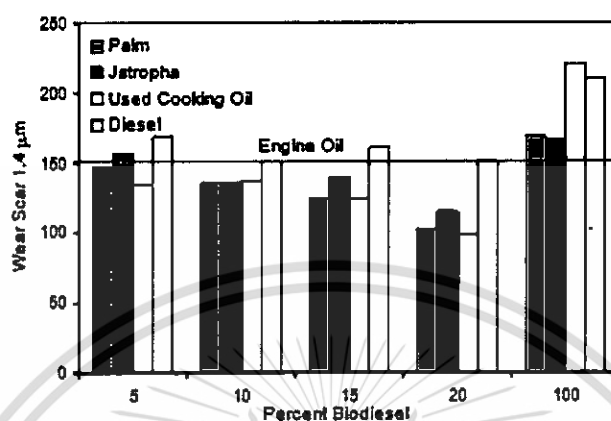


Figure 2.11 Wear scar of sample oils.

Kiatkong Suwannakij and his team [26] investigated biodiesel contamination in engine lube oil by using a high frequency reciprocating rig (HFRR) and Ball-on-Flat test. The results of HFRR showed in figure 2.11 that the wear scar of biodiesel 20 decreased when increasing the percentage of biodiesel in engine oil whereas diesel contamination resulted in the relatively constant wear scar of Palm and waste cooking oil biodiesel showed the most effect to reduce the wear, followed by Jatropha biodiesel. The WS 1.4 results seem to be contrary to the expectation that blending biodiesel should increase wear scar due to lower viscosity of biodiesel than engine oil.

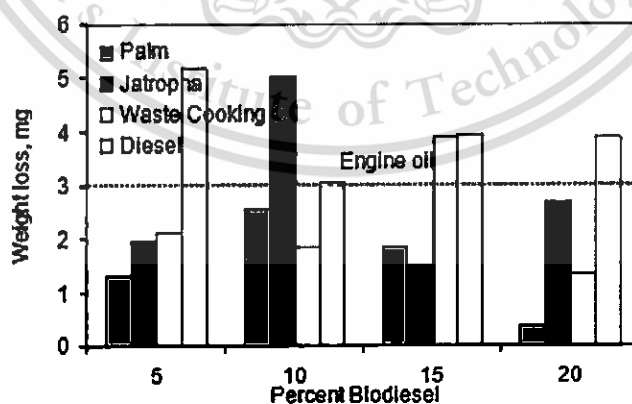


Figure 2.12 Weight loss of cylinder liner.

The cylinder liner was measured by weight scale. The weight losses of specimen which submerged under oil samples are shown in figure 2.12. Although the weight loss did not correlate with the percentage of fuels in sample oil, biodiesel tended to reduce the wear on the cylinder liner when they were blended with lubricant. The reduction of wear by adding biodiesel corresponds with the HFRR results. Palm biodiesel showed superior effect than those of *Jatropha* and waste cooking oil while diesel dilution increased wear.

The HFRR is the method to evaluate the sample oil lubricity, associated with the boundary lubrication. This means that fresh biodiesel improves the lubricity of oil dilution, because it contains more natural molecular species with boundary lubricating properties than mineral oils.

In order to clarify the effect of different biodiesel feedstock on lubricity, the compositions of biodiesel are required. Majority composition of palm biodiesel is palmitic (C16:0) which is a saturated fatty acid whereas *Jatropha* biodiesel composes of longer chain unsaturated oleic (C18:1) and linoleic acids (C18:2). Depending on the original sources, waste cooking oil presents the same composition as its source. Therefore, in this case for Thailand, palmitic (C16:0) should be the majority composition.

Referring to biodiesel composition, saturated fatty acids (palmitic) of palm and waste cooking oil biodiesel seem to improve the lubricity of engine oil for the current study. However, it is stated that unsaturated fatty acids and extended chain length could enhance lubrication. Hence, *Jatropha* should increase more lubricity. This discrepancy may be caused by the impurities in biodiesel such as monoglycerid and free fatty acid.

CHAPTER 3

Methodology

3.1 Sampling

In this research used synthetic SAE 0w-30 lubricating oil and oil filter were kept from real 1.5 liter diesel engine with difference mileage between 2,000 km to 20,000 km and lubricating oil age between 2,000 km to 10,000 km, the specification of engine and lubricating oil will show following;

Table 3.1 Engine specification

Engine parameter	Specification
Displacement volume	1,500 cc
Bore x Stroke	76.0 x 82.6 (mm)
Compression ratio	14.8
Maximum Power	105/4000 (hp/rpm)
Maximum Torque	280/1,500-2,500 (Nm/rpm)
Lubricant	SAE 0W-30

From table 3.1 Shows the specification of small diesel engine the compression ratio of this engine is 14.8 by using synthetic SAE 0W-30 are lubricating oil for lubricated system.

Table 3.2 Lubricating oil SAE 0w-30 specification

Properties	Specification
Viscosity @ 40 °C, (cSt)	44.5
Viscosity @ 100 °C, (cSt)	9.6
Viscosity Index	207
Oxidation, (Abs)	18.1
TBN, (mg KOH/g)	5.6

From Table 3.2 shows lubricating oil specification of synthetic SAE 0w-30, viscosity is 44.5 cSt at 40 °C and 9.6 cSt at 100 °C and total base number (TBN) is 5.6 Abs. These parameters will be decrease when the engine was used and lubricant system of engine will contaminate by air, fuel also soot. In case of oxidation and nitration will be increase.

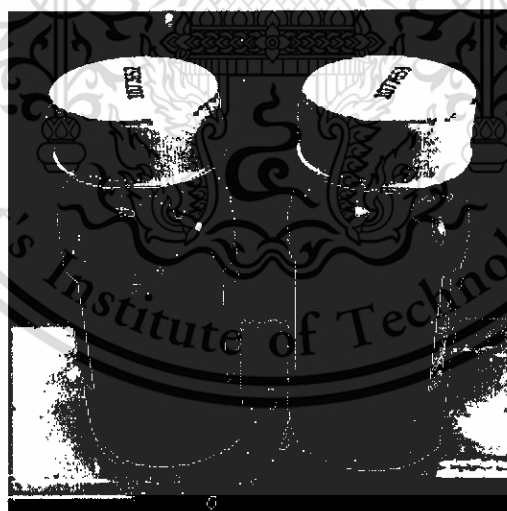


Figure 3.1 Sample of used lubricating oil compare with new lubricating oil

Figure 3.1 shows distinction of both lubricating oil the left hand side represent used lubricating oil and the right hand side represent new lubricating

oil. The color of used lubricating rather dark than new oil because of contamination of soot, fuel, water and chemical reaction.

Table 3.3 Mileage and Oils sample specification

Lubricating oil/car sample	Mileage (km)	Lubricating oil age (km)
1	1,980	1,980
2	3,975	3,975
3	6,275	6,275
4	8,468	8,468
5	9,993	9,993
6	11,237	11,237
7	19,046	9,362
8	19,500	9,800
9	19,613	9,448
10	19,779	9,811

From table 3 shows mileage and lubricating oil age of all samples. In the car number 1 has minimum mileage and oil age as 1,980 km. If observations of this mileage and oil age same data because the car number 1 is in run in process that mean this car was drove 1,980 km and the 1st time for change oil age. The car number 6 has maximum oil age and filters age but still in run in process like car 1 to car 5. Car number 7 to number 10 are 2nd oil change there aren't difference in mileage and oil age in this group. The maximum mile age is 19,779 km as car number 10.

3.2 Experimental procedures

3.2.1 Experimental procedures of used lubricating oil and oil filter

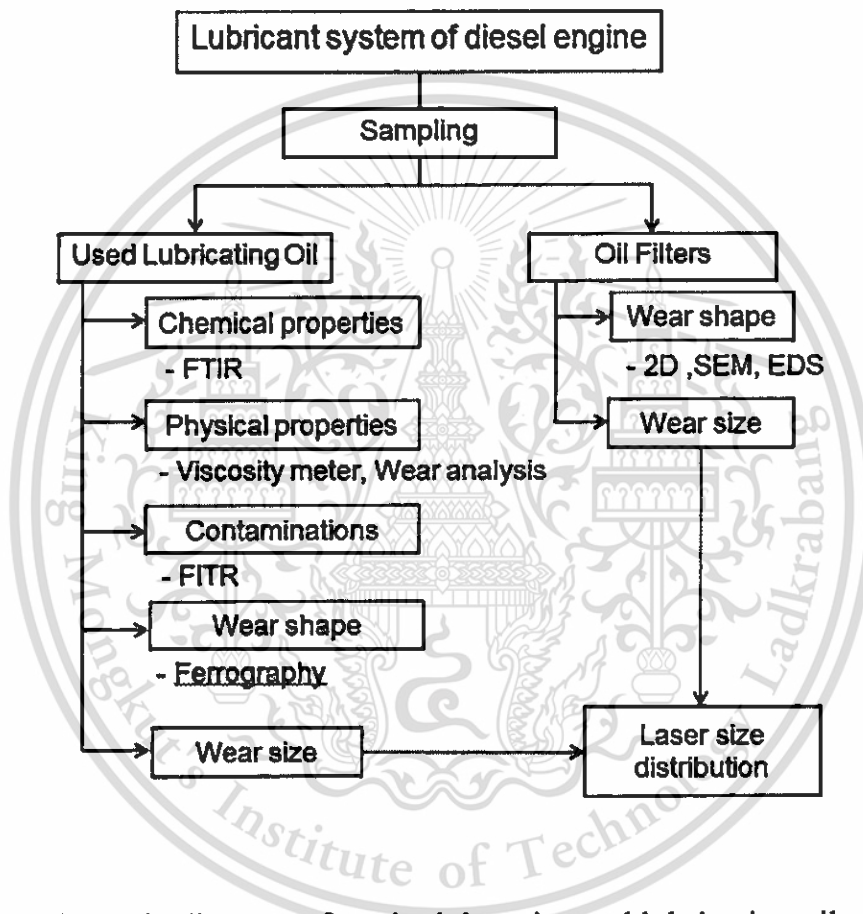


Figure 3.2 Schematic diagram of methodology in used lubricating oil and filter

Figure 3.2 shows the schematic diagram of methodology in used lubricating oil and filter which used in this research. There are two main tests the first test is used lubricating oil characteristic. In this part consist of chemical properties, physical properties, contamination and optical test. The second part is oil filter in this part have been test with optical test.

3.2.2 Experimental procedures of fuel contamination

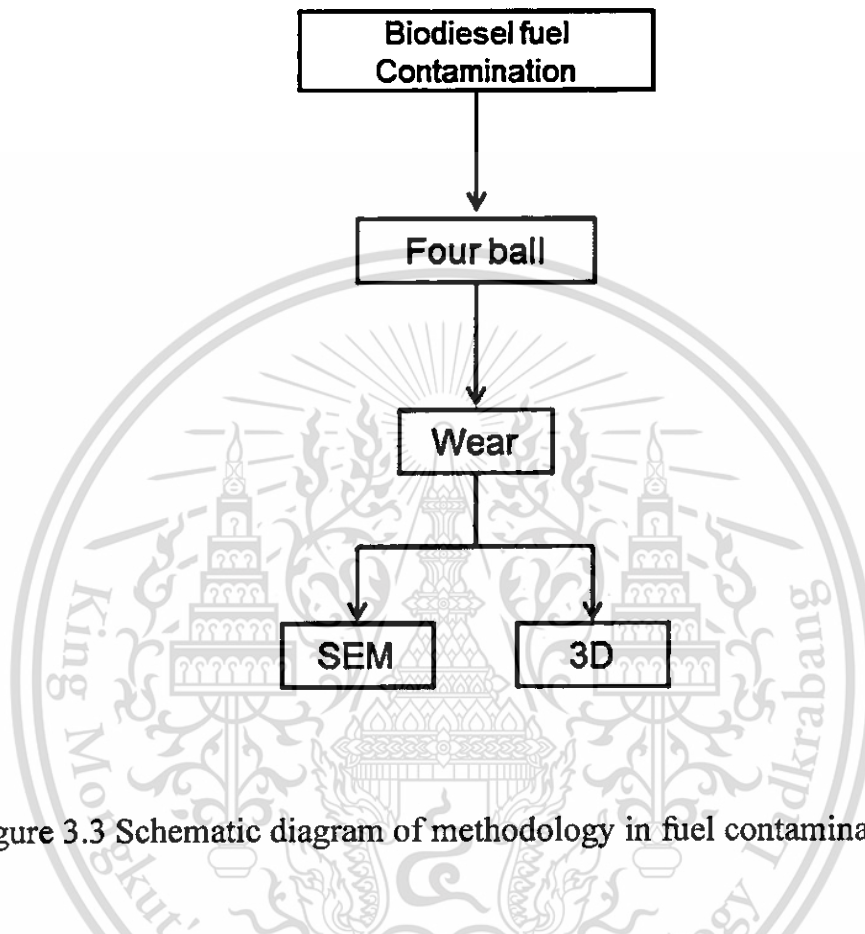


Figure 3.3 Schematic diagram of methodology in fuel contamination

Figure 3.3 shows the schematic diagram of methodology in fuel contamination which used in this section research. There are two tests in this section, the first test is physical properties and the second test is optical test. The study of friction, wear, and lubrication is of enormous practical importance, because the functioning of many mechanical, electromechanical, and biological systems depends on the appropriate friction and wear values. In recent decades, this field, termed tribology, has received increasing attention as it has become evident that the wastage of resources resulting from high friction and wear is greater than 6% of the gross national product.

3.2.2.1 Equipment of used lubricating oil characteristic test



Figure 3.4 Fourier Transform-Infrared Spectroscopy (FTIR)

Figure 3.4 shows Fourier Transform-Infrared Spectroscopy (FTIR) by Fourier Transform-Infrared Spectroscopy (FTIR) is an analytical technique used to identify organic (and in some cases inorganic) materials. This technique measures the absorption of infrared radiation by the sample material versus wavelength. The infrared absorption bands identify molecular components and structures. When a material is irradiated with infrared radiation, absorbed IR radiation usually excites molecules into a higher vibrational state. The wavelength of light absorbed by a particular molecule is a function of the energy difference between the at-rest and excited vibrational states.

The wavelengths that are absorbed by the sample are characteristic of its molecular structure. The FTIR spectrometer uses an interferometer to modulate the wavelength from a broadband infrared source. A detector measures the intensity of transmitted or reflected light as a function of its wavelength. The signal obtained from the detector is an interferogram, which must be analyzed with a computer using Fourier transforms to obtain a single-beam infrared spectrum. The FTIR spectra are usually presented as plots of intensity versus wavenumber (in cm^{-1}). Wavenumber is the reciprocal of the wavelength. The intensity can be plotted as the percentage of light transmittance or absorbance at each wavenumber. This research used the FTIR for test oxidation, nitration, total base number (TBN), percent of soot and fuel contaminate.

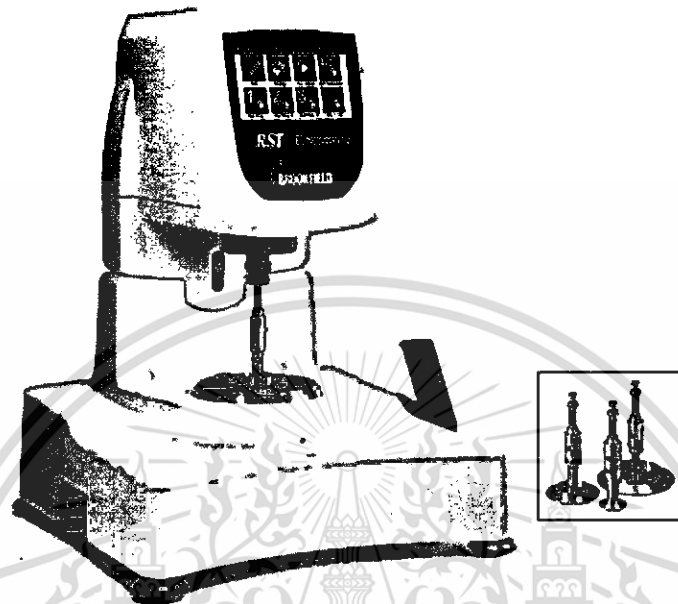


Figure 3.5 Viscosity meter

Figure 3.5 Shows viscosity meter by viscosity is a principal parameter when any flow measurements of fluids, such as liquids, semi-solids, gases and even solids are made. Brookfield deals with liquids and semi-solids. Viscosity measurements are made in conjunction with product quality and efficiency. Anyone involved with flow characterization, in research or development, quality control or fluid transfer, at one time or another gets involved with some type of viscosity measurement. There are many different techniques for measuring viscosity, each suitable to specific circumstances and materials. The selection of the right viscometer from the scores of instruments available to meet the need of any application is a difficult proposition.

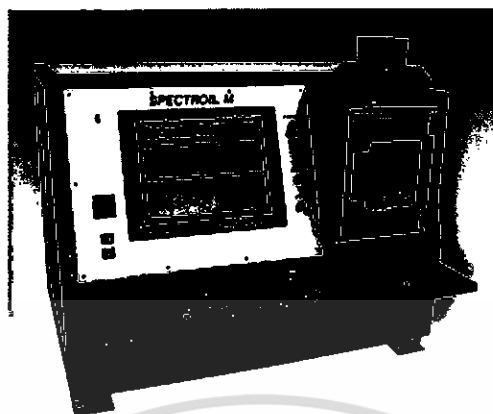


Figure 3.6 Wear analysis optical spectrometers

Figure 3.6 shows wear analysis optical spectrometers by the spectroil M/C-W is a compact, rugged, transportable and easy to use optical spectrometer designed specifically for wear particle analysis, contaminants and additives in lubricants, hydraulic fluids and coolants. They use the time-tested and reliable rotating disc electrode (RDE) technique to measure quantities of dissolved and suspended fine particles in natural or synthetic petroleum based products and coolants. The spectroil family of oil analysis optical spectrometers consists of the spectroil M/C-W for commercial oil analysis, the spectroil M/N-W for military applications and the spectroil M/F-LD for gas turbine and diesel engine fuel analysis. All versions of the spectroil M family are identical in hardware and differ only in their application and the way they are calibrated.

The spectroil M/C-W has become the standard instrument at most commercial oil analysis laboratories and machine condition monitoring programs that require the rapid wear particle analysis, contaminants and additives in lubricants. It fulfills the requirements of ASTM D6595 Standard Method for determination of wear metals and contaminants in used lubricating oil or hydraulic fluids by rotating disc electrode atomic emission spectrometry.

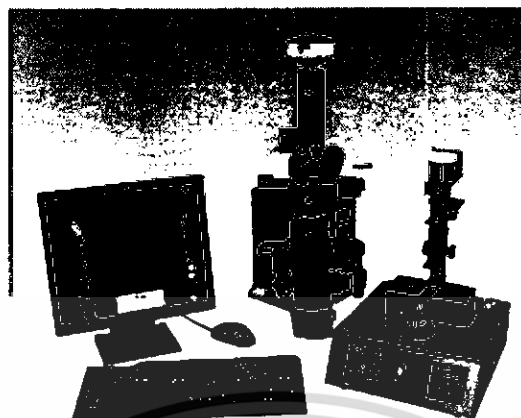


Figure 3.7 The spectroil analytical ferrography

Figure 3.7 shows the ferrography analytical in this equipment is a very useful and comprehensive analysis for trending and reporting. At a minimum, the user can obtain an analytical means of monitoring the wear condition; and, at the same time, accrue the necessary sample points required to establish a wear particle concentration baseline and retain the presence of non-magnetic particles for visual inspection and evaluation.

The advantage of ferrography over other preventive maintenance systems is its capacity to detect a broader range of types and sizes (0.1-500 microns) of wear particles. A micron is one millionth of a meter. Ferrographic analysis encompasses wear (metallic and non-metallic), contaminant (crystals, water, and organic and inorganic compounds), and lubricant (friction polymers) monitoring. Typical wear problems identified by ferrography; gear teeth wear through excessive load or speed, misalignments, fractures, rolling contact failure, water in the oil or poor lubricant condition, oil additive depletion, outside contaminants such as sand or dust, cam shaft and cylinder wall failure, oil filter failure.



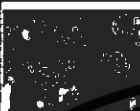
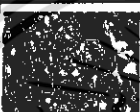


Actual Photos	Description	Examples of Causes
 Cutting Wear	Long, curled strips of metal	Normal Machine Wear
 Spherical Wear	Small Spheres	Contamination or rolling contact fatigue failure (Bearings, cams, and gears.)
 Red Oxide Particles	Red particles aligned in magnetic field	Water in the oil or poor lubricant condition
 Bearing Wear	Flat (1 micron thickness) particle with perforations	Rolling Contact Fatigue
 Molybdenum Disulfide	Nonferrous particle, gray in color with many shear planes.	Solid lubricant additive in system
 Friction Polymers	Amorphous materials which pass transmitted light	Excessive load or stress

Figure 3.8 Type and cause of wear

There are several type of wear that were generate from the engine, in figure 3.8 shows type and cause of wear, industrial application of ferrography entails the non-interruptive machine condition monitoring of heavily used lubricated mechanical systems. Hence, an operational baseline can easily be established by sampling every 50-500 hours of operation (approximately every one to three months, depending on system criticality), and used for quantitative trending analysis. Any anomalies in the wear particle concentration, especially in the generation rate of large particles (>20 microns), is symptomatic of the onset of failure. For consistent results and accurate trending, lubricant samples are taken from the same places in the system each time. The method of sample extraction assures that the lubricant samples contain a representative selection of wear particles. The samples are then ferrographically analyzed both quantitatively and qualitatively.

3.2.2.2 Equipment of oil filters test

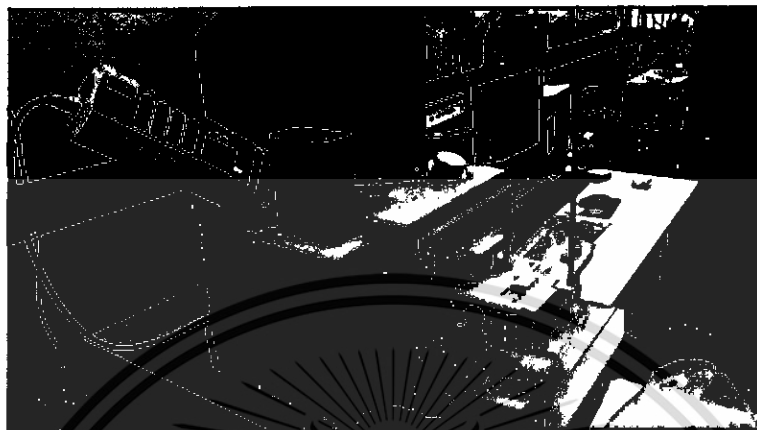


Figure 3.9 Scanning electron microscopy (SEM)

Figure 3.9 shows Scanning electron microscopy (SEM) is a method for high-resolution imaging of surfaces. The SEM uses electrons for imaging, much as a light microscope uses visible light. The advantages of SEM over light microscopy include much higher magnification ($>100,000\times$) and greater depth of field up to 100 times that of light microscopy. Qualitative and quantitative chemical analysis information is also obtained using an energy dispersive x-ray spectrometer (EDX) with the SEM. The SEM generates a beam of incident electrons in an electron column above the sample chamber.

The electrons are produced by a thermal emission source, such as a heated tungsten filament, or by a field emission cathode. The energy of the incident electrons can be as low as 100 eV or as high as 30 keV depending on the evaluation objectives. The electrons are focused into a small beam by a series of electromagnetic lenses in the SEM column. Scanning coils near the end of the column direct and position the focused beam onto the sample surface. The electron beam is scanned in a raster pattern over the surface for imaging. The beam can also be focused at a single point or scanned along a line for x-ray analysis. The beam can be focused to a final probe diameter as small as about 10 Å.

CHNS Elemental Analysers

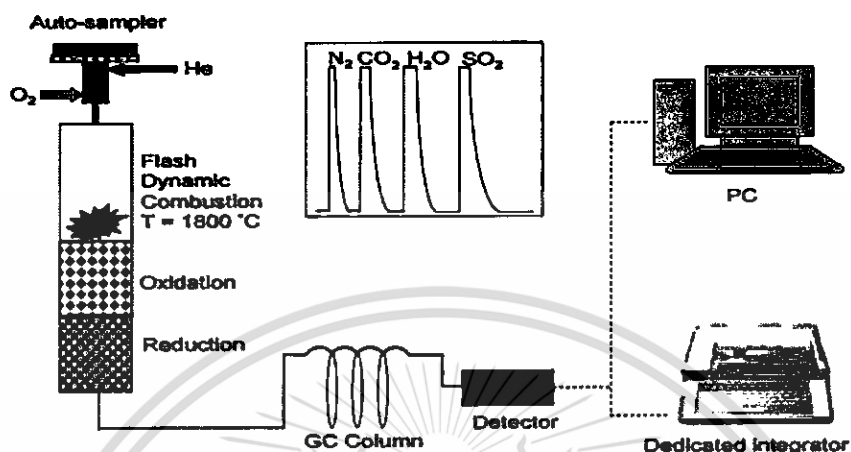


Figure 3.10 Schematic diagram of CHNS element analyzers

Figure 3.10 CHNS elemental analysers provide a means for the rapid determination of carbon, hydrogen, nitrogen and sulphur in organic matrices and other types of materials. They are capable of handling a wide variety of sample types, including solids, liquids, volatile and viscous samples, in the fields of pharmaceuticals, polymers, chemicals, environment, food and energy. The analyzers are often constructed in modular form such that they can be set up in a number of different configurations to determine, for example, CHN, CHNS, CNS or N depending on the application.



Figure 3.11 The laser diffraction granulometer and associated dispersion units

Figure 3.11 shows the laser diffraction granulometer and associated dispersion units, laser diffraction technique. It is used in a wide range of applications to give accurate measurement of particle size. The method is usually applicable for a particle range from 0.1 to 2,000 μm depending on the setup of the system, A particle volume fraction from 0.001 to 1.0 vol.% can normally be measured. The laser diffraction technique is based on Mie scattering developed by Gustav Mie.

In order to solve for a spherical particle Mie assumed that the particle has to be isotropic, in effect no difference in which direction the incident beam strikes the particle. He also assumed that the waves of the incident light were of constant frequency, constant amplitude and parallel.

The particle size distribution of all samples were performed with the laser diffraction granulometer and associated dispersion units This laser diffraction particle size analyzer is designed to measure particle sizes ranges from 0.02 to 2000 μm by using a blue (488.0 μm wavelength LED) and red (633.8 μm wave length He-Ne laser) light dual-wavelength, single-lens detection system. The light energy diffracted by the dilute suspension circulating through the cell is measured by 52 sensors.

3.2.2.3 Equipment and condition of fuel contamination test

Table 3.4 Percent of fuel contamination in four ball test

Lubricants	% Lubricant	Fuel Type	Fuel
SAE 0W-30	100 %	-	-
SAE 0W-30	98 %	B7	2 %
SAE 0W-30	98 %	B20	2 %
SAE 0W-30	98 %	B50	2 %
SAE 0W-30	98 %	B100	2 %

Table 3.4 shows percent of fuel contaminate with new lubricating oil by in this section test were test in four ball tester to know in case of wear and friction with fuel contamination issues on the rise and high pressure injection systems becoming less tolerant of this contamination monitoring of fuel quality has become an important effect to know when we decide to used biodiesel as main fuel.

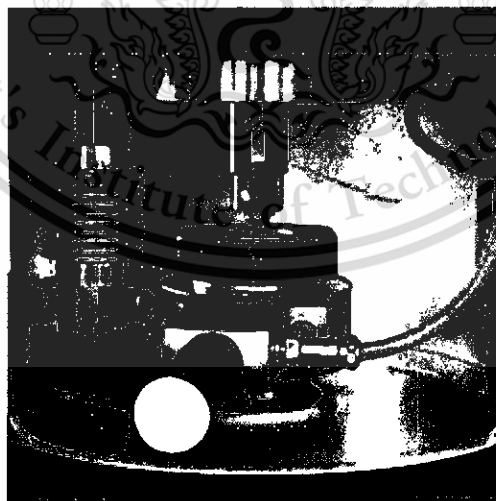


Figure 3.12 Four ball tester

In this section have test with four balls to know, what is the effect of fuel for tribology in case of wear and friction also the four ball tester was used in this part shows conditions test;

Table 3.5 Condition test of four ball tester

Parameter	Specification
Rotational speed	1200 rpm
Load	392 N
Duration per load	60 min
Temperature	75°C

Figure 3.12 shows the four ball tester is an excellent development and quality check instrument for developers and users of lubricants and additives. The unique sample configuration of three bottom balls and one top ball makes a very stable and a repeatable contact in-turn, allowing tests results to be very repeatable.

The Four Ball Tester can be used to determine Wear Preventive properties (WP), Extreme Pressure properties (EP) and friction behavior of lubricants. The wide acceptance of test results of the four ball tester makes it an excellent choice to benchmark products. It is a good choice for R&D due to its relatively inexpensive samples and ability to produce quick and repeatable results.

A rotating steel ball is pressed against three steel balls firmly held together and immersed in lubricant under test. The test load, duration, temperature and rotational speed are set in accordance with standard test schedule. In Wear Preventive (WP) tests – also called Anti Wear (AW) tests – the average scar diameter on the bottom three balls is reported.

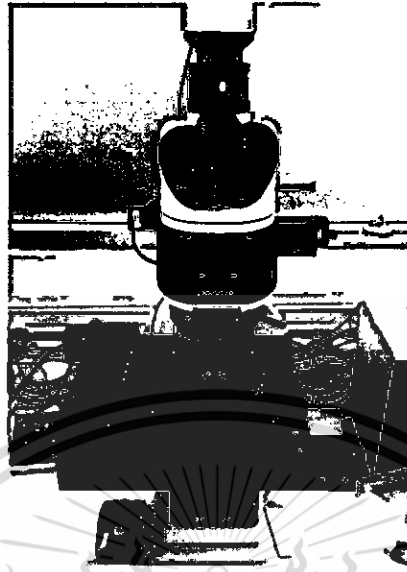


Figure 3.13 3D optical microscope

Figure 3.13 shows the 3D optical microscope for finding the size of wear scars that generate from a four-ball tester. A 3D optical microscope has become a versatile tool for measuring areal surface topography and texture in a wide variety of industries. A key factor in the growth of industrial applications has been hardware and software implementations that streamline and simplify its use, particularly for technician-level users.

Quantitative 3-D surface metrology measuring surface topography as well as the size and shape of microscopic surface features – is vital to many industries. In applications including coated glossy paper, painted automobiles or molded plastic parts, surface texture affects cosmetic appearance and, hence, perceived value. But in many cases, such as with automobile engine components, medical implants or high-brightness LEDs (HB-LEDs), the surface topography can be absolutely critical to proper part functioning.

CHAPTER 4

RESULTS AND DISCUSSIONS

4.1 Physical properties of used lubricating oil

4.1.1 Viscosity

Any changes in viscosity of the lubricating oil are undesirable in an engine as it affects the lubrication effectiveness. Two major factors are mainly responsible for lubricant oil viscosity changes, (i) formation of resinous products because of oil oxidation, evaporation of lighter fractions, depletion of anti-wear additives and contamination by insoluble compounds tend to increase viscosity and (ii) moisture addition, fuel dilution and shearing of viscosity index improvers tend to reduce the oil viscosity. The kinematic viscosity of lubricant oil was evaluated at 40 °C and 100 °C. The decrease in viscosity was compared with initial lubricating oil viscosity during the new period of engine operation. The figure 4.1 and 4.2 show that the decrease trend of viscosity in used the lubricating oil decreases. The average viscosity at 40°C is 36.68 centistokes and average viscosity at 100 °C is 8.06 centistokes.

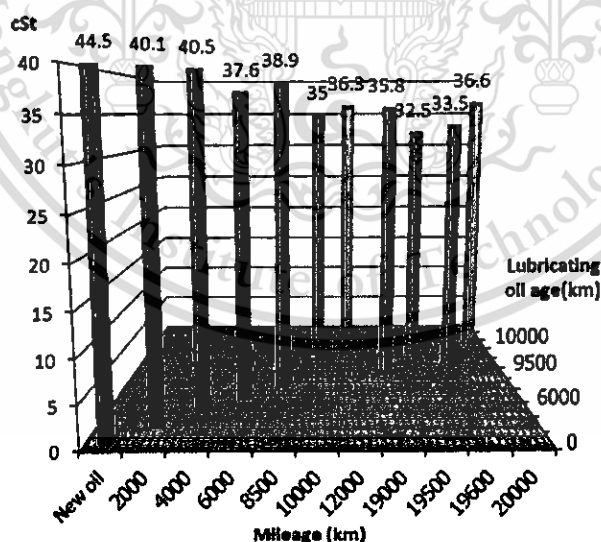


Figure 4.1 Impact of viscosity at 40°C on mileage and lubricating oil age

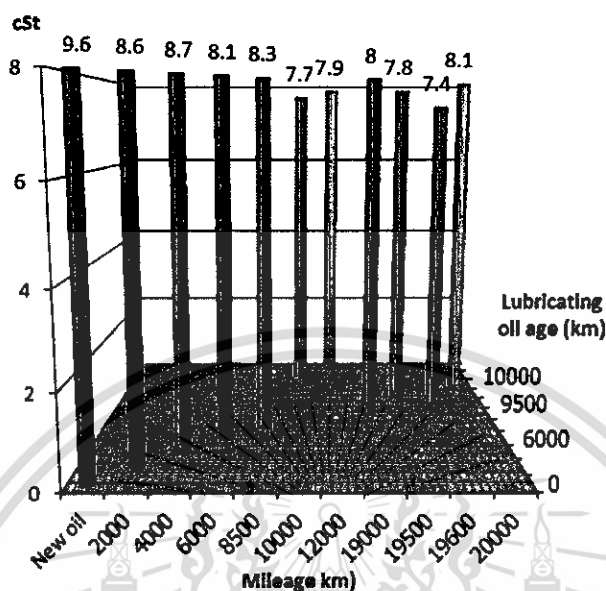


Figure 4.2 Impact of viscosity at 100°C on mileage and lubricating oil age

4.1.2 Wear

Wear is a progressive loss of material over time. Although engine wear often appears to be a local component-level issue, it needs to be considered in the early stage of engine system design because the wear life time of the component is closely related to system loading. For example, peak cylinder pressure affects the wear life of the valve seat, the piston assembly and the engine bearings.

The wear of engine components usually undergoes three stages: (1) a running-in period where the rate of change is high; (2) a stabilized normal running period where a steady rate of wear is maintained; and (3) a wear-out failure period where a high rate of wear leads to rapid failures. Generally, there are four common types of wear leads in the engine: abrasive wear, adhesive wear, fatigue wear and corrosive.

The Figure 4.3 shows impact of wear on mileage and lubricating oil age from the result when the mileage increase the wear will be increase because of when the engine run many part of engine touch together. The highest wear is on the sample 9 with 281.2 ppm, the average of this wear is 121.62 ppm.

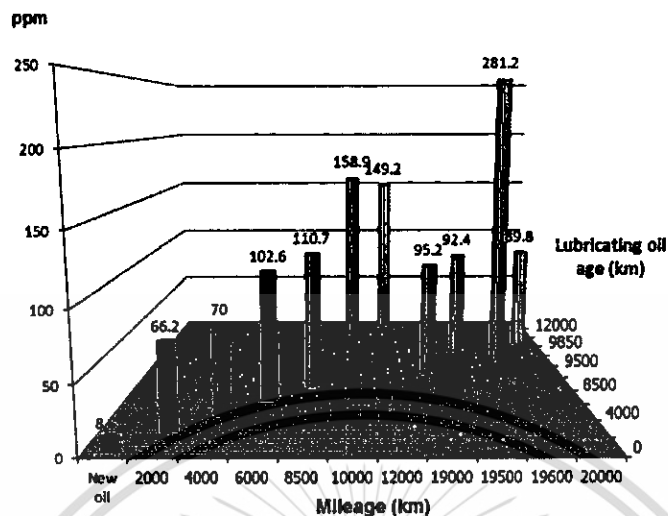


Figure 4.3 Impact of wear on mileage and lubricating oil age

4.1.2.1 Wear debris

Wear debris analysis is well-known in condition monitoring of tribosystems. The conditions of machine operation are related to the process of wear debris generation and finally to their morphology. Thus, classification of debris in different morphological classes provides valuable information on the current state of a tribosystem. Metallic wear debris are differentiated by their morphology (shape, texture and color) into several classes, e.g. rubbing, cutting, spherical, laminar, fatigue chunk and severe sliding wear particles. It has been found that each type has its own generation mechanism involving a specific wear process.

For instance, cutting wear particles are produced by the penetration, plowing or cutting of mating bodies. The presence of severe sliding wear particles in a machine usually indicates a lubrication problem, as a result of lubricant film breakdown. Initially, the morphology of wear particles was examined visually by a trained expert. Nowadays, advances in computers and image recognition make automatic evaluation of the particle morphology possible. It may be characterized by a set of numerical features, and then appropriate classification methods can be used for wear particle identification. There are numerous papers that have considered wear debris morphology quantitatively (a good review), but practically all of them deal with problems of shape and texture.

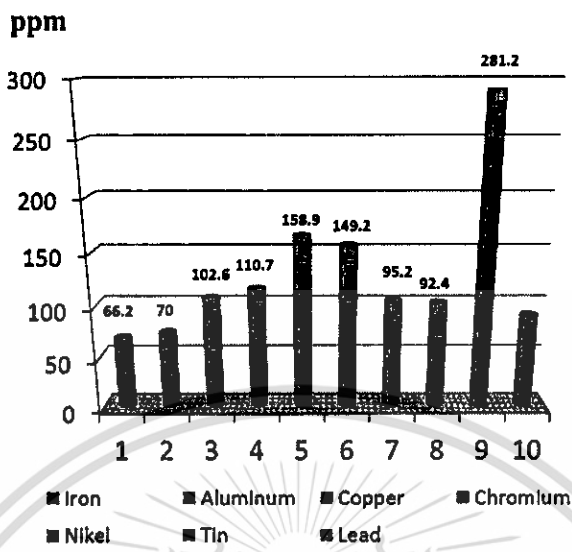


Figure 4.4 Total metal wear

The figure 4.4 show total particle metal for earch sample highest metal particle is in sample 9 in 281.2 ppm by over the average more than 2 times from this graph the result shows when the mileage and lubricating oil age increase the total metal and wear debris increase form the cat sample 9 hace highest metal particle. The one reason maybe is the wear debris in engine oil are catalyze to in crease the wear particle in the diesel engine.

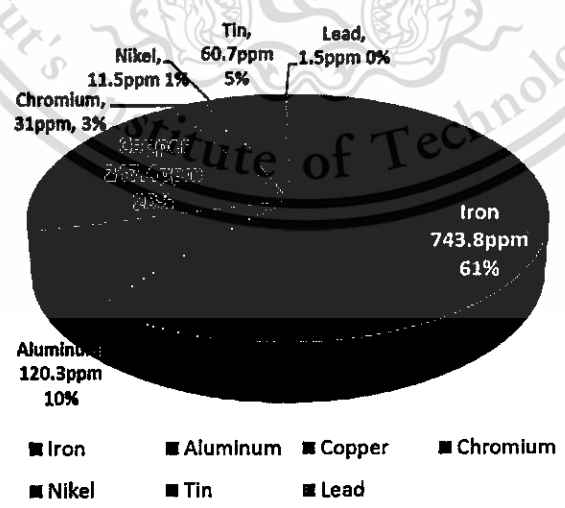


Figure 4.5 Wear element

Wear debris in diesel engine, lubricating system wear particle was washed and suspended in the lubricating oil. The analysis of the concentration metallic particle in lubricating oil provides sufficient information and prediction about the wear rate, element source and engine condition. The common metallic components found in lubricating oil after the engine operation were iron (Fe), copper (Cu), aluminum (Al) and lead (Pb). The Iron is the most element wear that found in wear debris possibly caused by the wear of cylinder liner, piston rings, valves, gears, shafts, bearing, rust and crankshaft from the experiment all Iron element found in 61% by 743.8 ppm.

The concentration of Cu is depicted in the green color. There are 247.4 ppm by 20% of all wear debris. The most common sources of copper concentration are bearing and bushing. Based on the wear debris analysis it was observed that the highest level of copper concentration produced by diesel engine.

Aluminum (Al). The Al concentration indicated piston wear or ingested dust. The red color shows the Al concentration over the engine running time. There is 120.3 ppm, 10% aluminum of all wear debris.

Ferrography is a technique for analyzing the particles present in fluids that indicate mechanical wear. Ferrography provides Microscopic Examination and Analysis of Debris (particles) found in lubricating oils. These particles consist of metallic and non-metallic matter. The metallic particle is a wear condition that separates different size and shapes of metallic dust from components like all type of bearings, gears or coupling (if lubricated in path). Non-metallic particle consists of dirt, sand or corroded metallic particle. Analytical ferrography is among the most powerful diagnostic tools in oil analysis in tribology.

When implemented correctly it provides a tremendous information on machine under operation. Yet, it is frequently excluded from oil analysis programs because of its comparatively high price and a general misunderstanding of its value. Performance may be improved through proper filtration of oil. Clean oil lubrication is always more effective. Adopting approach of oil replacement is expensive. A rapid centrifuged and/ or magnetic separator cleaning system helps cost cutting and disposal of used oil, as well. Ferrography also helps improving filtration efficiency and frequency for oil cleaning systems.

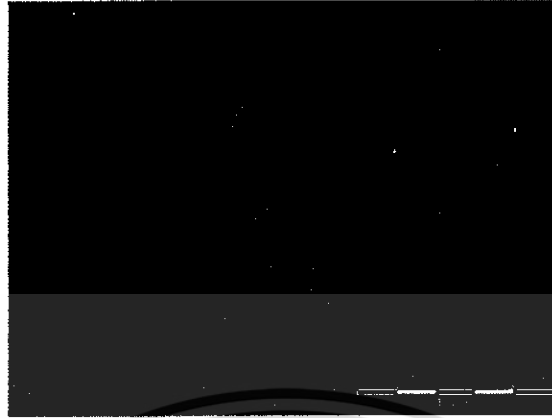


Figure 4.6 New oil SAE 0w-30

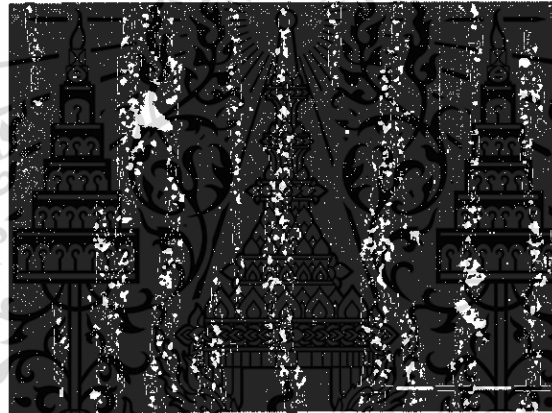


Figure 4.7 Wear debris in used lubricating oil

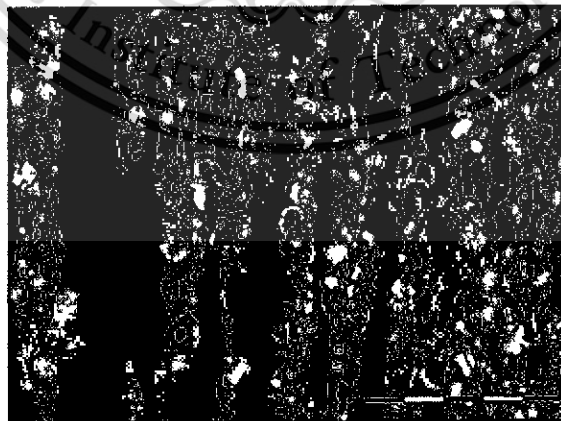


Figure 4.8 Wear debris in used lubricating oil

This material is reserved for educational use only, not allowed for commercial use.

Forbidden to modify the content, and cite the document when use.

The figure 4.6 shows the image from ferrogram it looked clear because this figure no metal contamination from engine but in figure 4.7 shows the normal rubbing wear the size around 5 μm to 20 μm . This is a normal particle the found in normal diesel engine generated.

In figure 4.8 show abnormal wear debris this figure from the car sample 9, not only wear metal was found in this sample but also found the long curl strips in the middle position in this sample. The main cause of long curl strips wear come from misalignment of soot contaminate.

Wear particle	Contaminant form	Percent (%)	Size (μm)
Normal rubbing wear		65	5-20
Fatigue bearing wear		20	5-30
White Metal		5	5-10
Black Oxide		5	5-15
Dirt and Dust		5	10-40

Table 4.1 Type and size of wear contaminate in used lubricating oil.

In used lubricating oil there are several wear type such as normal rubbing wear, fatigue bearing wear, white metal, black oxide, dirt and dust, the size of wear shows in table 4. More than 50 percent of wear is normal rubbing wear there are 65 percent in diesel engine which size 5 to 20 μm . The percent of each wear show in figure 4.9.

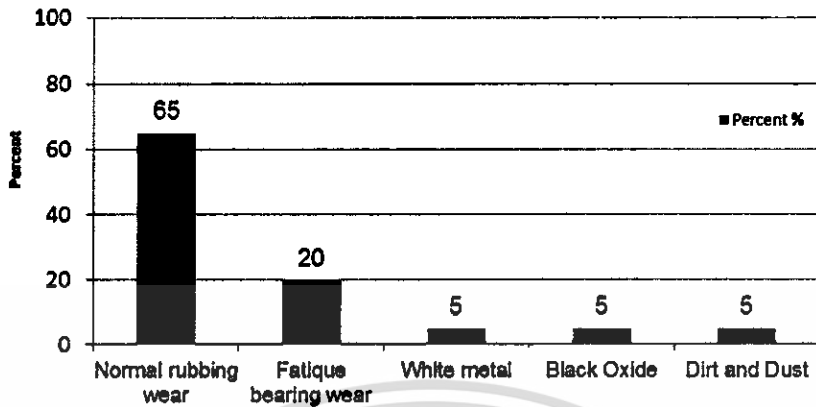


Figure 4.9 Type of wear in used lubricating oil.

4.2 Chemical properties of used lubricating oil

4.2.1 Oxidation

The presence of oxidation residuals in oil is an increasing problem in various oil systems. The problem is enhanced by the trend of down-sizing oil reservoirs, increasing oil temperatures and prolonging oil change intervals. A high temperature caused oil degradation instigates the formation of gasses in the oil; a phenomenon utilized in the electrical power generation industry to trace faults in oil filled power transformers. In transformers oil oxidation is caused by the oxygen present in the oil. The surface of the paper-isolation material (thin strips of cellulose wound around the copper cores) in a transformer are often coated by a brownish substance, particularly in old transformers.

Figure 4.10 shows the impact of oxidation on mileage and lubricating oil age, the result show that when the mileage and lubricating oil age increase the oxidation rate increase when compare with the new oil we will see the oxidation rate 18.1 Abs in new oil because every mineral oil have hydrocarbon also they can easy to reac with oxygen with the air but the trend also increase the one reason because mostly of oxidation come from the incomplete combustion and leak to the lubricating oil between piston and combustion chamber.

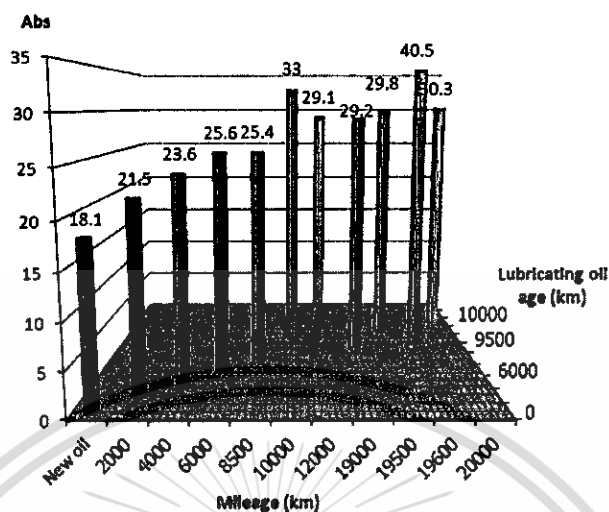


Figure 4.10 Impact of oxidation on mileage and lubricating oil age.

4.2.2 Total base number (TBN)

Total Base Number (TBN) is one of the neutralization number specifically used to measure the alkalinity reserve remaining in the lubricant. It is an indication of lubricant's ability to neutralize corrosive acids that formed during the engine operation. Higher TBN value gives low concentration of free acids of lubricating oil of oil samples. More ash content in lubricating oil will obstruct engine oil filters, excessive fuel consumption and shortens the engine life. Ash content of lubricating oil samples are measured as per ASTM D 482 standards. It is defined as the ratio of the mass of ash to the total mass of the lubricating oil.

The figure 4.11 shows the impact of total base number on mileage and lubricating oil age, in new lubricating oil the TBN is 5.6 mgKOH/g the total base number decrease when mileage and lubricating age increase the average of TBN of this experiment is 3.15 mgKOH/g the minimum TBN is in car sample 5 and number 9 respectively.

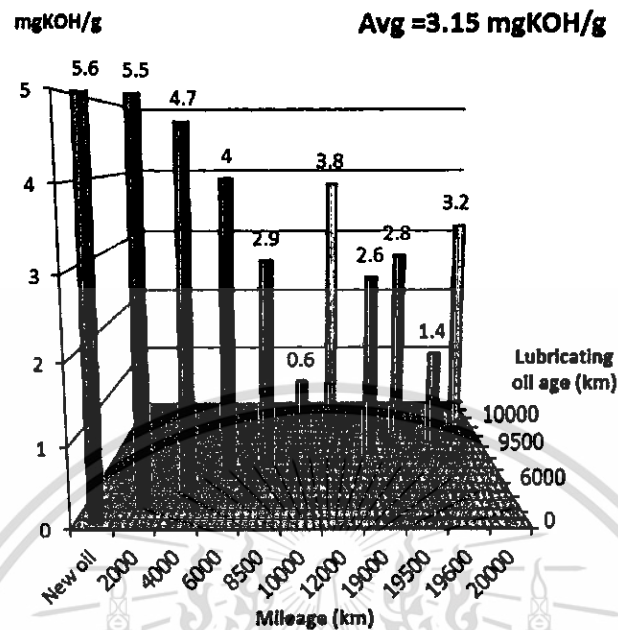


Figure 4.11 Impact of oxidation on mileage and lubricating oil age.

4.3 Contamination in used lubricating oil

Diesel engine lubricant contamination is a major cause of engine component wear, leading to loss of engine performance and life. In addition, contamination accelerates breakdown of the engine lube oil, reducing its useful service life. Contaminant particles the size of or larger than the dynamic lubricant oil films separating moving component surfaces cause a major portion of diesel engine wear. The size of these harmful particles is 20 microns and smaller.

4.3.1 Soot

Internal combustion engines produce soot as a result of incomplete fuel combustion. Ideally, complete combustion in a cylinder would only produce carbon dioxide and water, but no engine is completely efficient. Soot occurs because of the way that fuel is injected and ignited, soot formation occurs more commonly in diesel than in gasoline engines. The fuel and air mixture in diesel engines typically do not mix as thoroughly as they do in gasoline engines. This creates fuel-dense pockets that produce soot when ignited.

While the majority of soot easily escapes through the exhaust, some gets past the piston rings and ends up in the oil. Form figure 4.12 shows the percent

of soot contamination, when the mileage increase and the oil age increase the amount of soot will be increase in new lubricating oil there is none of soot contamination, the average of soot contamination is 0.69 % Weight. The highest soot contaminate at the car number 9 by 1.01 % it's is a lot of soot contamination.

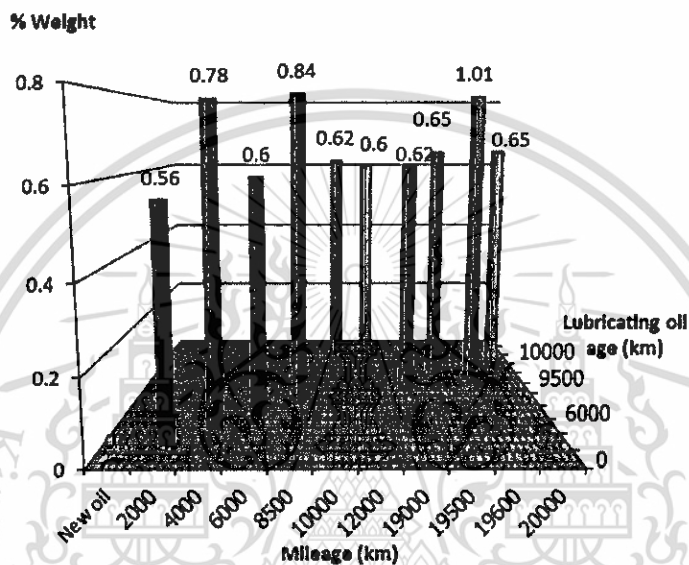


Figure 4.12 Impact of soot on mileage and lubricating oil age.

In part of wear debris and wear contamination the sample number 9 is highest metal wear debris that from the long curl strips was found in ferrogram. In the section the soot contaminate in car sample 9 from filtergram was found a lot of soot distribution in filtergram test. Filtergram technique is used to detected non-metallic particle which consists dirt, sand or corroded metallic particles as shown in figure 4.13 black points are distribution of soot appeared in grean chart, their size varies between $0.1 \mu\text{m}$, these contamination in lubrication oil could be generated by combustion blow effect as discussed earlier.

Figure 4.14 shows the soot distribution from car sample 9 when compare filtergram test with each other car this car has the contamination of soot and soot distribution.

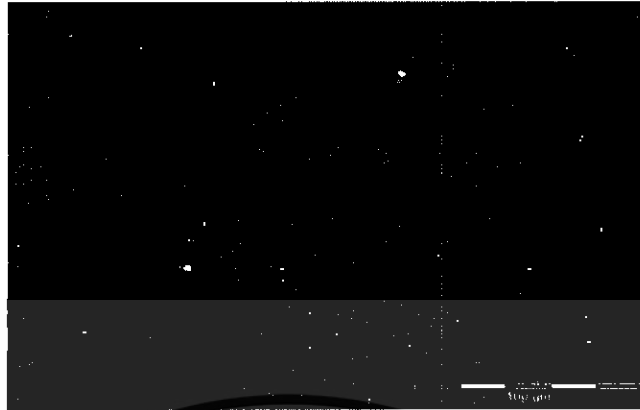


Figure 4.13 Soot distribution in lubricating oil of car number 6.



Figure 4.14 Soot distribution in lubricating oil of car number 9.

4.3.2 Fuel

Detecting fuel contamination in engine oil by any method is difficult because fuel and base oil differ in terms of molecular weight, boiling temperature range, and aromatic compound content. The IR absorption method enables the examination of the aromatic compounds in engine oil, and thereby, the determination of fuel residue.

The difference in aromatic compound content between engine oil and fuel reflects fuel residue. In this work, therefore, the spectra of the fresh engine oil around 800 cm^{-1} was taken as the baseline for detecting possible fuel residue. The absorption intensity of the fresh engine oil was measured, and the change in its absorption spectrum was determined as it was being diluted. The

IR absorption value of each absorbance spectrum was derived for all the collected samples and plotted for comparative analysis.

Figure 4.15 shows fuel contamination of all samples by the average of fuel contamination in small diesel engine is 2 % it means this sample are normal of fuel contamination, because if fuel contamination more than 5% the engine have to overhaul. From the result show that when the mileage and lubricating oil age increase percent of fuel contamination will be increase.

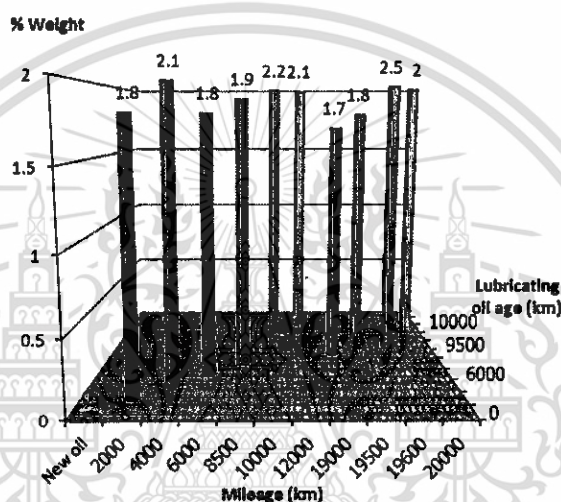


Figure 4.15 Impact of fuel contamination on mileage and lubricating oil age.

4.4 Oil filter

Oil does a number of important things in an engine. It helps cool the engine by transferring heat; seals the gap between piston rings and cylinder walls; absorbs contaminants; and suspends soot particles that are the residue of combustion. Contaminants can be organic such as fungus and bacteria from the air. Inorganic particles can be a host of things, including dust that is sucked into the engine during running.

If left by it-self for very long the oil would become saturated with the byproducts of combustion. It would carry minute, harsh particles that would wear the oil pump's machined components as well as the bearing surfaces in the engine. It always has done so, but in early engines the oil was changed so often (due to burning and poor oil quality) that no filtration was required. Later on

some manufacturers put mesh screens in the oil path to grab bigger particles but this was not very effective.

The outside of the filter is a metal can with a sealing gasket that allows it to be tightly held against the engine's mating surface. The base plate of the can holds the gasket and is perforated with holes around the area just inside the gasket. A central hole is threaded to mate with the oil filter assembly on the engine block. Inside the cans is the filter material, most frequently made from synthetic fiber. The engine's oil pump moves the oil directly to the filter, where it enters from the holes in the perimeter of the base plate.

Dirty oil is passed (pushed under pressure) through the filter media and back through the central hole, where it re-enters the engine. Typically, filters have two media types: primary and secondary. The primary media can stop particles as small as 25-30 microns (human hair is 65-70 microns in size) and the secondary media can stop particles as small as 5-10 microns. "Full flow" filters only utilize primary media.

Secondary media limits flow, so the use and placement of it is highly restrictive in filters. Secondary media, when incorporated in a filter, operate in a partial-flow way. Inside the filter a small portion (1-10%) of the oil is directed through the secondary filter and the rest flows through the primary.

The longer a filter is on the car the more particles are suspended in the media, until a point is reached where oil can't flow through it. A bypass valve is incorporated in the engine's filter assembly to handle that situation, but once the oil bypasses the filter no cleaning action takes place.

A few filter manufacturers are extolling the virtue of placing magnets in the filters to trap metal particles. They claim that magnetic forces stabilize the oil molecules; that the longer the magnetic field is in place the greater the benefit; that fuel mileage is increased; oil consumption and emissions are decreased; and engine power is increased. None of these claims are true and several are impossible.

4.4.1 2D optical microscope

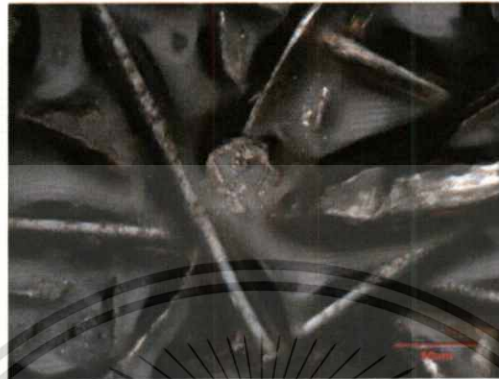


Figure 4.16 Particle before clean in fiber



Figure 4.17 Particle after clean in fiber

From the figure 4.16 and 4.17 show the particle was trapped in filter whereas around 30-100 μm , there are from the oil filter and look like the normal rubbing wear that showed in ferrography technique from previous part.

4.4.2 Scanning electron microscope (SEM) and energy dispersive x-ray spectrometer (EDS)

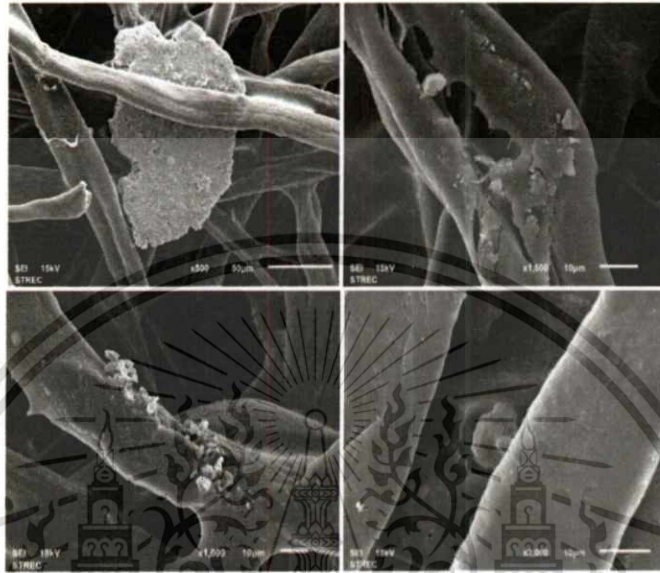


Figure 4.18 Particle in fiber of oil filter from SEM image

From figure 4.18 four type of particles from fiber in oil filter was found their size around 1 to 100 μm . The top left and bottom left look like particle of metal they can rend in to the fiber. The top right and the bottom right look like soot or carbon composite they cannot rend the fiber unlike the left picture.

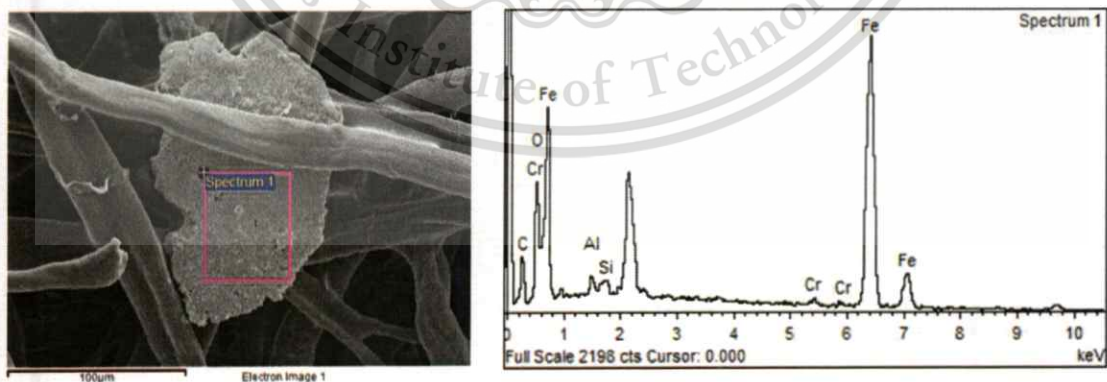


Figure 4.19 Agglomerate metal particles from EDS test (A)

This material is reserved for educational use only, not allowed for commercial use.

Forbidden to modify the content, and cite the document when use.

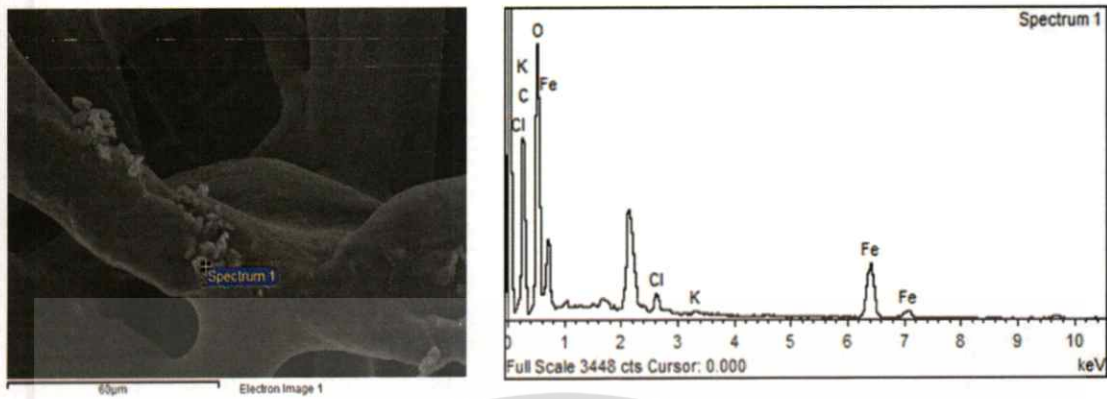


Figure 4.20 Small metal particles from EDS test (B)

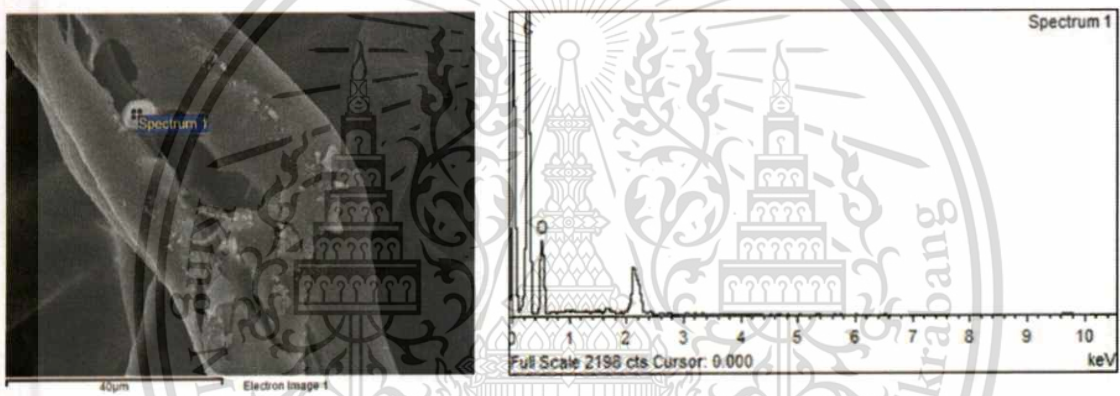


Figure 4.21 Soot particles from EDS test (C)

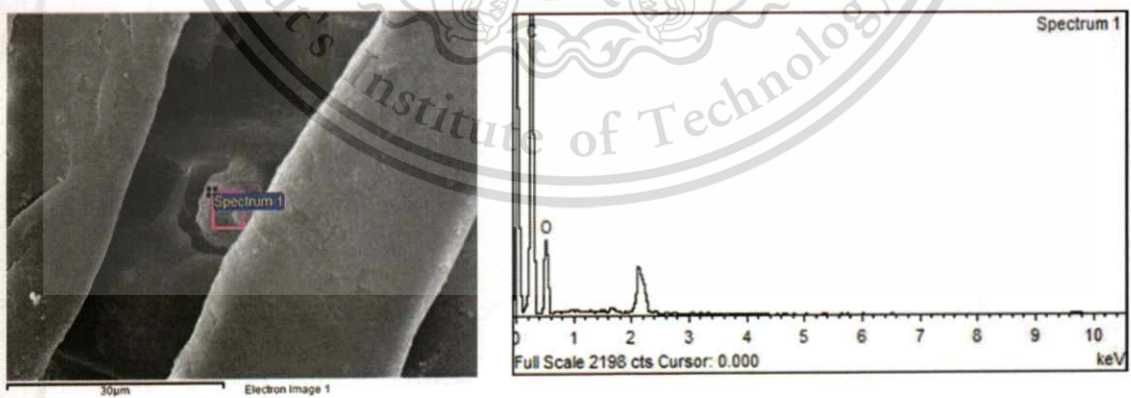


Figure 4.22 Carbon composite particles from EDS test (D)

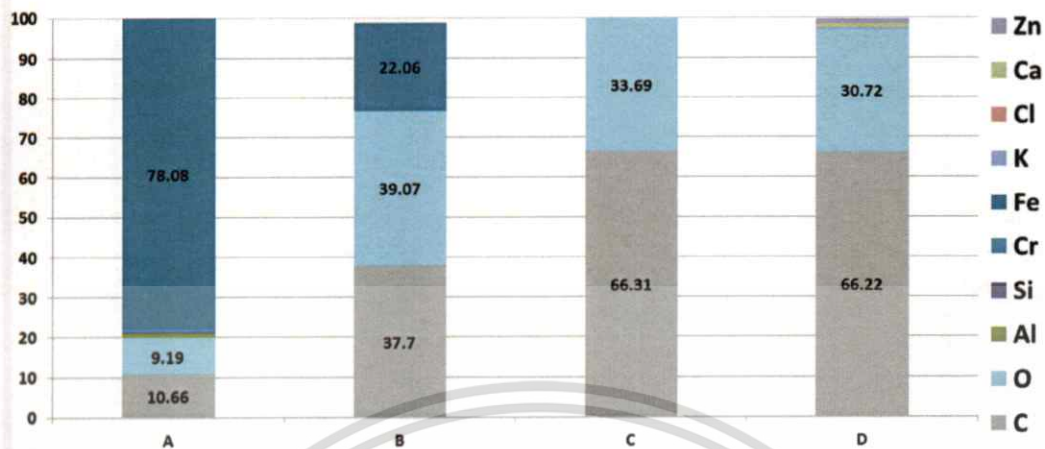


Figure 4.23 Element components in each particle

Scanning electron microscope showed wears debris, which has been measured in range of 0.5 μm to 100 μm and divided in to two main size groups. Debris diameter ranges from 0.5 μm to 5 μm , shown in figure 4.20, were measured using 1000X magnification scale while diameter ranges from 5 μm to 100 μm , shown in figure 4.19, were measured with 500X magnification scale. Groups of metal wear debris were identified from energy dispersive X-ray microanalysis, that consisting O39%, C37%, Fe 22% Ca1.5% and K 0.37% by weight. Figure 4.19 showed the largest group of wear debris, size is 80 μm the following components were present, Fe 78.08 %, O 10.66%, Al 0.92%, Cr 0.77% and Si 0.37 % respectively.

From the constituent of element can observed, these group of wear debris efficacy can be inserted and rend in to the fibers of filter. If a large number of wear debris metal group are found in used lubricating oil that indicates reduction in life of lubrication oil and which are very critical to cause abrasion wear in the engine. Main cause of adhesive wear occurs when the boundary layer of lubricant film between the asperities of two opposing surfaces is displaced. However other wear debris was generated by the high pressure in the combustion chamber, which is termed as combustion blow by. The wear debris by combustion blow directly affect to cylinder liner and piston rings resulting deterioration on sealing efficiency consequently, the deposition of soot in rings due to incomplete combustion also reduces the sealing efficiency.

The second group of particles pack that showed in figure 4.21 and 4.22 shows, particle size distribution is very wide. Particles measuring 5 μm to 20 μm , identified by EDX contains two third of carbon and one third of oxygen. As noticed from Figure 4.21, higher percentage of carbon in those nodular

shapes suggests the presence of soot particles in tested lubricant. Additionally, those flat shaped particles as shown in figure 4.22 consists of 79.32% of carbon and 20.68% of oxygen, also seems like soot particles although the shape is different. The variation in shape may be due to the variation in carbon and oxygen percentages. The primary soot particles contains 98 percent carbon by weight and typically spherical in shape. While most are only around 0.03 μm size, they often clump together to form larger particles. Individual soot particles pose little risk to engine parts, but clumps of soot can cause damage.

4.4.3 Particle distribution

To precision measure and identify the particle size distribution of lubricating oils and oil filters from five samples, laser diffraction technique was used for this experiment to detect size of particle. Figure 4.24 showed particles which appear in lubricating oils and oil filters. In used lubricating oil, the smallest size 0.1 μm and 23.3 μm . Particle distribution size of oil filters from five samples which size 1 μm to 100 μm were found. In figure 4.25 red line represent the average size of particles that existing in lubricating oils from five samples, the average of particles from lubricating oils is 2.7 μm . There are 2 peak of this blue line, the highest peak was 3.16 μm which 5.52 % and the second peak is 0.68 μm . which 4.38%. From the size distribution that showed in highest peak expected to be small wear debris group with reference to result from SEM. The second peak expected to be soot that from filtergram technique in figure 4.25 or even the small particle as second peak perhaps it could be dust, sand, oxidation emulsion, water and fuel because these are many factors which contaminate within the lubricating oils.

In figure 4.25, the red line represent particle distribution was emerged on that sample the highest peak in 4.3 μm which 6.2% and the second peak 0.68 μm size which quantity is 3.5%. The blue colors showed particle distribution size of filters from five samples, from graph 1 μm to 100 μm were found. Blue line represent the average size of particles from five samples is 18.54 μm . The most 14.7 μm size appeared 6.94 % by volume. At the biggest size is 100 μm appeared in 0.02 % within sample 4 in figure 4.24.

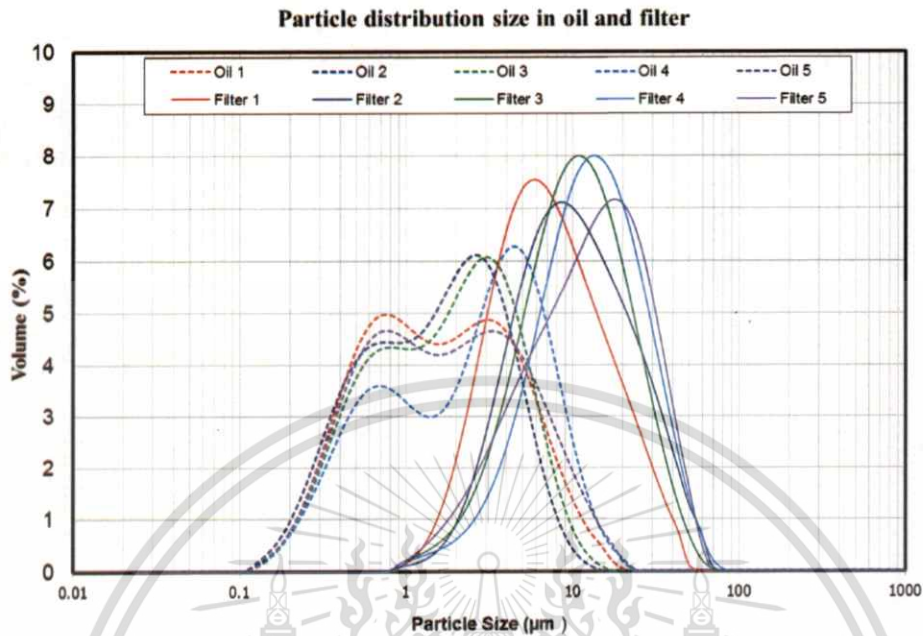


Figure 4.24 Particle distribution size of used lubricating oil and oil filter all samples.

From the result image from SEM can establish absolutely confident by these large particles are group of metal wear debris. Lubricating oil contamination causes wear debris which generates more contamination. This process is the chain reaction of wear. This proceeds by internal wear generating fresh wear debris; by internal wear opening dynamic sealing surfaces, allowing contaminant ingress from the environment, and by lubricating oil breakdown generating varnishes, sludge, decrease oil life and increase worse chemical reaction with in oil.

In Figure 4.25 the red line represent the average particle distribution size of five sample their average size is 2.7 µm. Dashed red line represents cumulative frequency of particles size of lubricating oils, show that since 0.1 µm until 1µm size was found in 36% by volume and since 1 µm to 10µm is 62% by volume this indicate that the mostly size of particle distribution in the lubricating oil is 1 to 10 µm. The second part is particle size distribution in filters, blue line represents the average particle distribution size of five sample their average size is 18.54 µm. Dashed blue line represent cumulative frequency of particles size of filters, 1 µm to 10µm size is 38% by volume and 10 µm to 100 µm size was found within 62% mean that mostly particle size in filter is 10 µm to 100 µm.

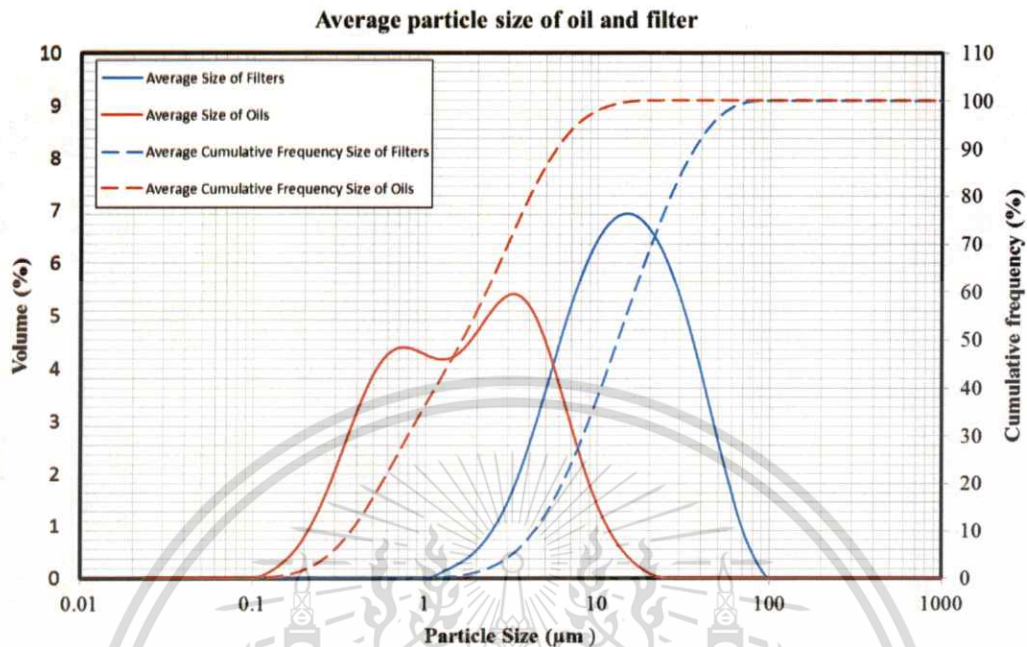


Figure 4.25 Average particle size in used lubricating oil and oil filters.

Figure 4.25 the intersection of red line and blue line indicates that filter can capture particle larger than 1 µm, however filtration efficiency of filter appears better for particles larger than 4 µm. The trend of red line suggests particles size of used lubricating oil start to decrease after 4 µm.

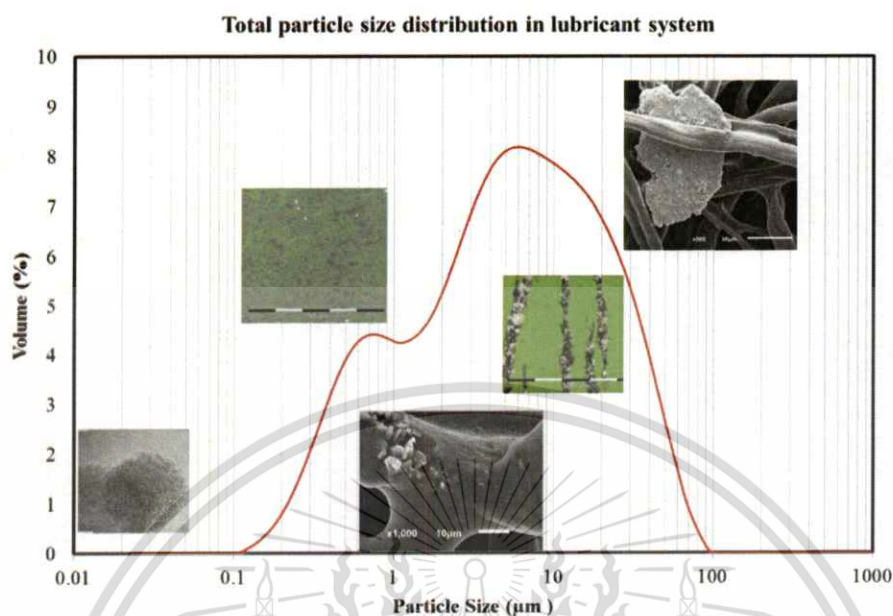


Figure 4.26 Size of particle from lubricating system of diesel engine.

Figure 4.26 red line represents total particle distribution size of all samples and type of particle that found from SEM and ferrography technique, that means, in the lubricant system of these five sample can generated particle size from $0.1 \mu\text{m}$ to $100 \mu\text{m}$ there are different distribution size around thousand times, by the most size of particle of this lubricant system can generate is between $4 \mu\text{m}$ to $6 \mu\text{m}$.

Particle less than $0.1 \mu\text{m}$ is soot PM 10. Particle more than $0.1 \mu\text{m}$ is soot that generated from incomplete combustion and leak in to the lubricating oil by combustion blow by process. Particle between $1\text{-}5 \mu\text{m}$ is metal wear debris to make sure from the result of SEM. Particle size $5\text{-}40 \mu\text{m}$ from ferrogram and filtergram method can identify difference wear such as normal rubbing wear, fatigue bearing wear, white metal, black oxide and dust. The biggest particle was found whereas $100 \mu\text{m}$ in filter this particle is metal wear in SEM.

4.5 Biodiesel fuel contamination

Table 4.2 Palm biodiesel specification.

Fuel	Viscosity at 40°C (cSt)	Carbon (%)	Hydrogen (%)	Oxygen (%)
Diesel (B0)	3.22	85.99	14.01	0
Commercial diesel (B7)	3.26	85.39	13.91	0.65
B20	3.42	83.59	13.61	2.6
B50	3.784	80	13.01	6.5
B100	4.53	77	12	11
SAE 0w-30	44.5	95	5	0

Biodiesel has a major advantage over petroleum diesel, since, it is derived from renewable sources it is a clean burning fuel that does not contribute to the increase of carbon dioxide, being environmentally friendly Biodiesel is an oxygenate, sulfur-free and biodegradable fuel, and its content of oxygen helps improve its combustion efficiency Therefore, fewer greenhouse gases such as carbon dioxide are released into the atmosphere.

Biodiesel has positive performance attributes such as increased cetane, high fuel lubricity, and high oxygen content Since, biodiesel is more lubricating than diesel fuel, it increases engine life and it can be used to replace sulfur, a lubricating agent, that when burned, produces sulfur oxide; the primary component in acid rain.

Palm biodiesel is an oleaginous tropical plant, has the highest oil productivity per unit of land on earth. In terms of its usage, palm oil has various uses as a food, (oils, margarines, bread, mayonnaise, feeds, ice cream, cookies etc.), in industry (soap, lubricants, detergents, plastics, cosmetics, rubber etc.), in steel making, the textile industry, pharmacology etc.

Table 4.2 shows palm biodiesel specification of this experiment, from table there not only viscosity of biodiesel B100 has highest but also amount of oxygen is in biodiesel 100 on the other hand in diesel fuel is none of oxygen component and lowest viscosity.

4.5.1 Four ball tester

The Four-Ball testing is one of the tribological test methods. This test method can be used to determine the relative wear preventive properties of lubricating fluid in sliding contact under the prescribed test conditions. No attempt has been made to correlate this test with balls in rolling contact.

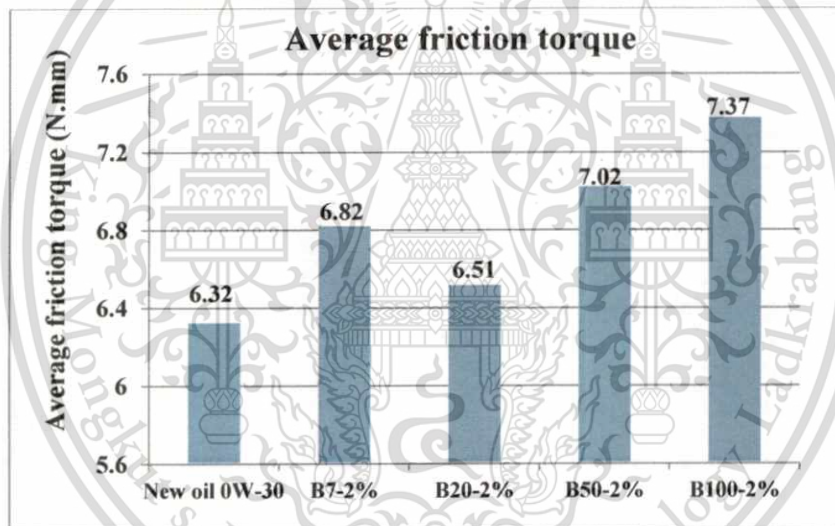


Figure 4.27 Impact of biodiesel fuel contamination to friction torque.

Figure 4.27 shows impact of biodiesel fuel contamination to friction torque the highest friction torque is 7.37 N.mm which in B1002%, minimum friction torque is 6.32 N.mm in pure lubricating oil 0w-30. In biodiesel B7-2% has average wear scar higher than pure lubricating oil and B20-2% but less than B50-2% and B100 2%.

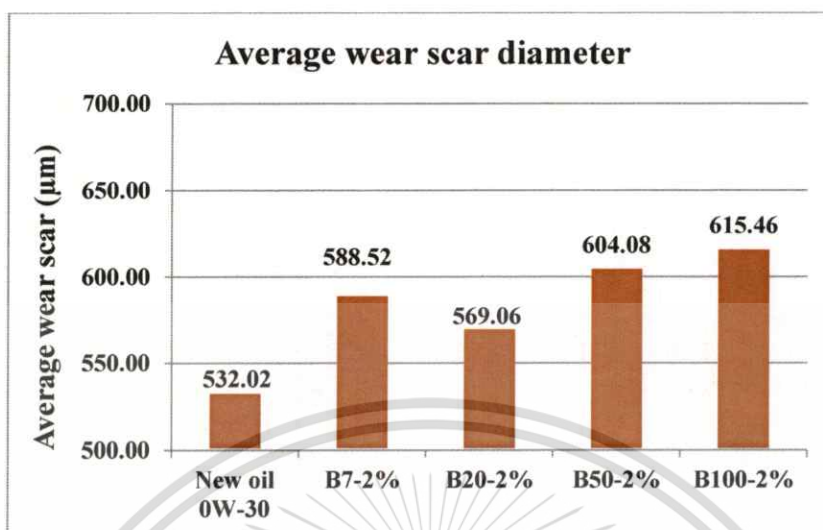


Figure 4.28 Impact of biodiesel fuel contamination to wear scar.

Figure 4.28 shows Impact of biodiesel fuel contamination to wear scar, from both figure, In B100-2% has highest wear scar by wear scar form B100-2% is $615.45\mu\text{m}$ by lowest wear scar is on pure lubricating oil.

Both result of average wear scar and friction torque, there are interesting in B20-2% because they made lower friction and wear scar than another % of biodiesel fuel contamination.

4.5.2 3D optical microscope

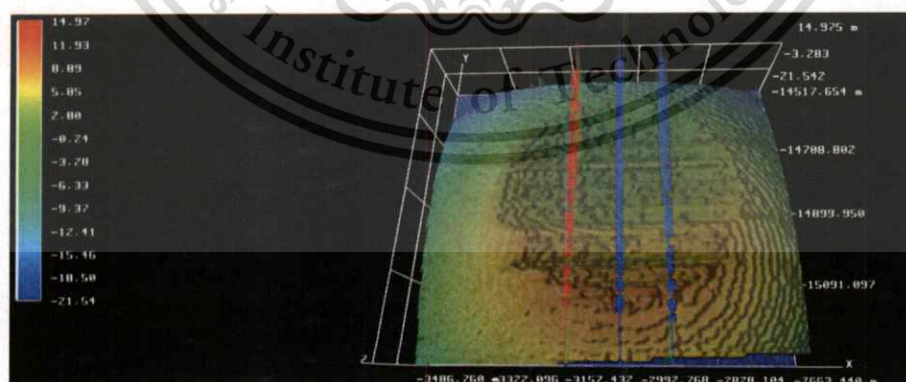


Figure 4.29 Deep scars in each sample from 3D microscope.

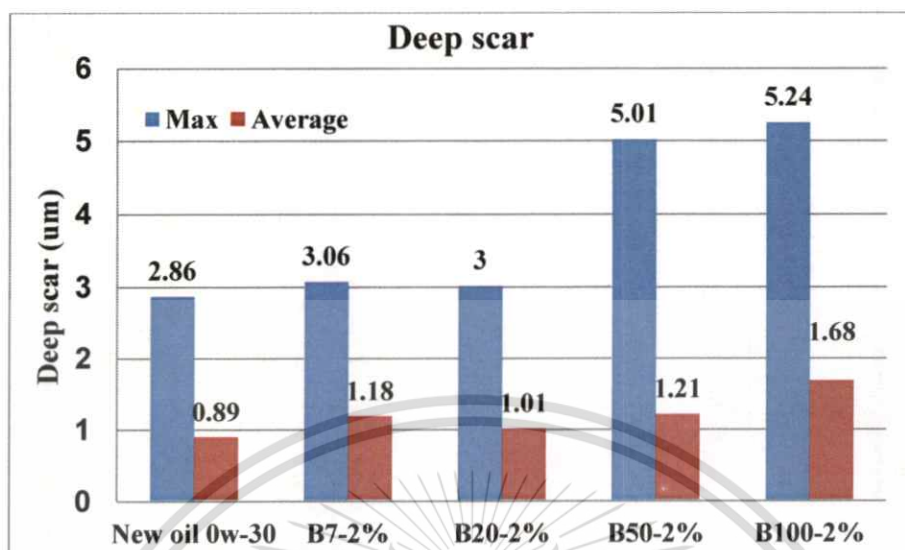


Figure 4.30 Average deep scars in each sample from 3D microscope.

Figure 4.30 show the average and maximum value of deep scar of steel ball after test from four ball tester, the results show that biodiesel B100-2% not only has highest average deep wear scar but also has highest maximum deep scar on the other hand the lowest deep scar and their average value is on pure lubricating oil at 2.86 and 0.89 μm respectively. There are interesting in Biodiesel B20-2% because it's has average wear scar lower than biodiesel B7-2%, B50-2% and also B100-2%.

4.5.3 Biodiesel fuel model blend with lubricating oil

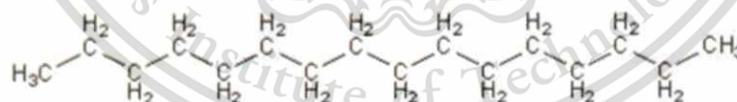


Figure 4.31 Typical diesel molecules.

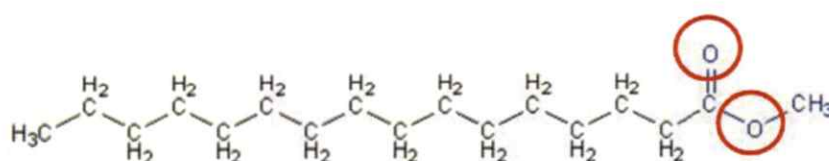


Figure 4.32 Typical biodiesel molecules.

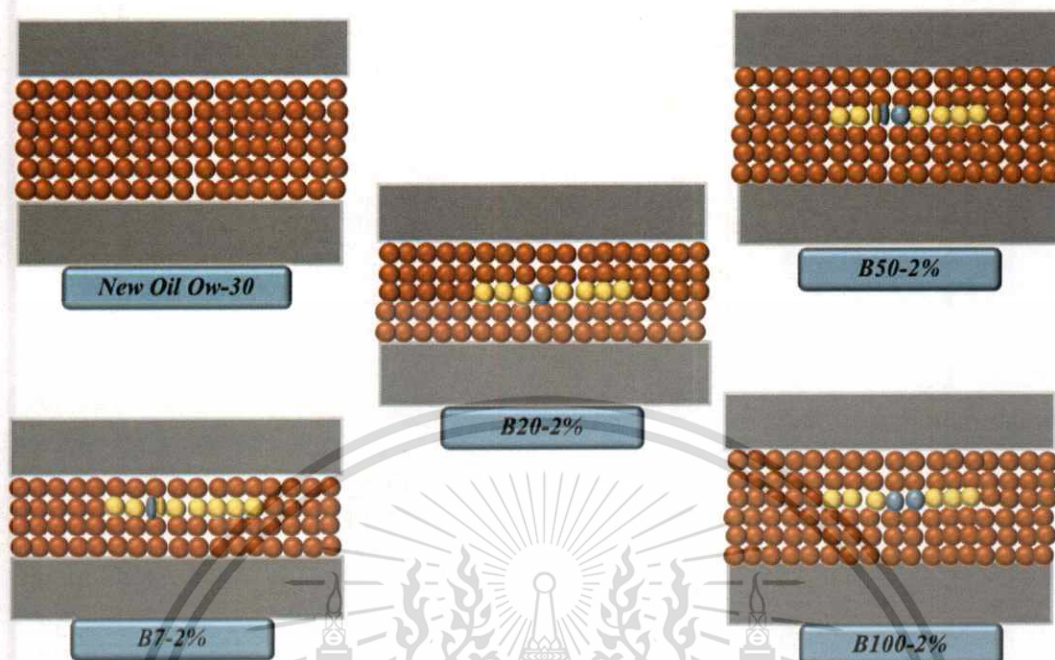


Figure 4.33 Model of biodiesel fuel blend with lubricating oil.

From interesting result of friction torque and wear scar of biodiesel fuel contamination, when compare all of fuel contamination percent of biodiesel B20-2 %, built minimum wear scar. The main reason of this result because the typical diesel molecules difference from typical biodiesel molecules in biodiesel the structure of molecule including with oxygen that showed in figure 4.31 and 4.32.

By the way viscosity and percent of oxygen in biodiesel, viscosity whereas biodiesel B100-2% is 3.78 that showed in table 4.2 on the other the hand the amount of oxygen in B100-2% is highest percent.

Although B100-2% has highest viscosity but B100-2% also have percent molecule of oxygen in structure of fuel. The effect of oxygen of this experiment not only can generate the oxidation reaction in lubricating oil and reduce life time of them but also cause to reduce the structural strength of lubricating oil so if we observe from the figure 4.33 when percent of biodiesel increase the viscosity will be increase when compare only blend fuel but that mean percent of oxygen also increase so after blend biodiesel fuel with lubricating oil amount of oxygen will be increase follow percent of biodiesel.

4.5.4 Wear scar from SEM image

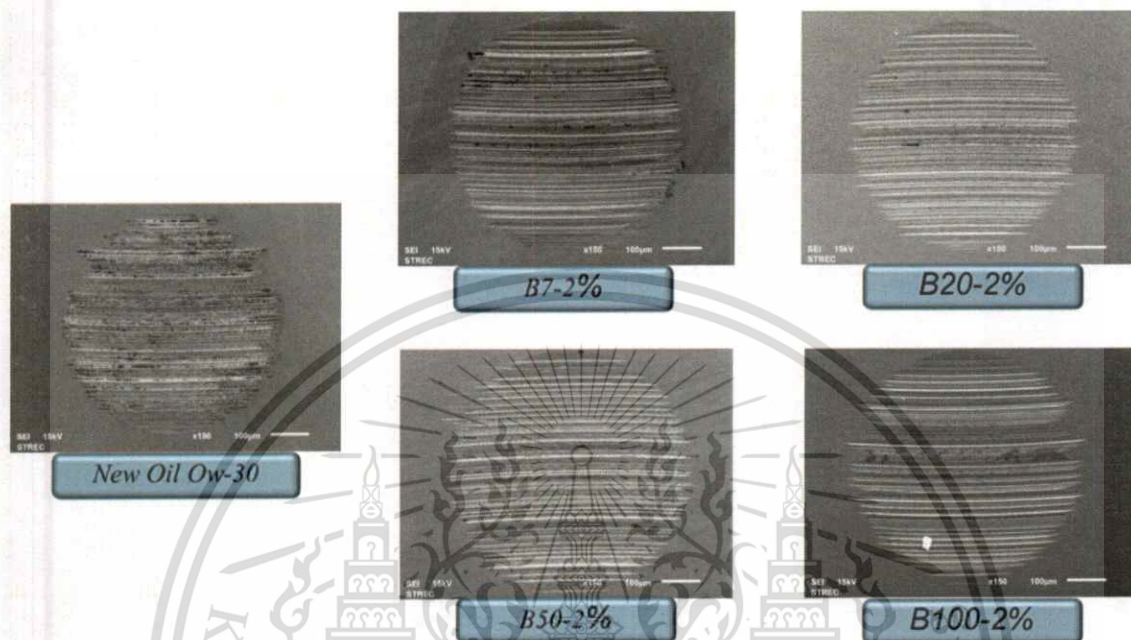


Figure 4.34 Wear scar in each blend biodiesel fuel of steel ball from SEM images with magnificent 150 times.

From figure 4.34 shows wear of steel ball from four ball tribotester in case of pure lubricating oil if we compare with another steel balls, we can observe that it is quite smooth. When we compare wear scar on steel ball contaminated with biodiesel fuel, most show slick wear scars whereas B20-2%. The most clearly wear scars are on steel ball B100-2% where not only abrasive wear but also fatigue wear is generated.

4.5.4.1 Abrasive wear

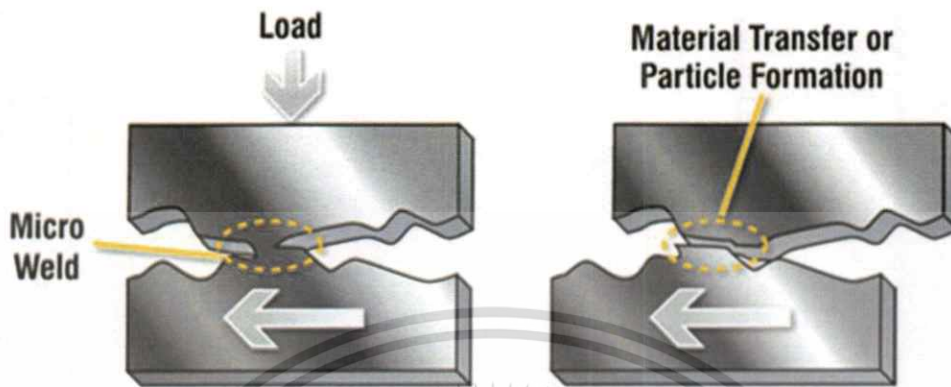


Figure 4.35 Mechanism of abrasive wear.

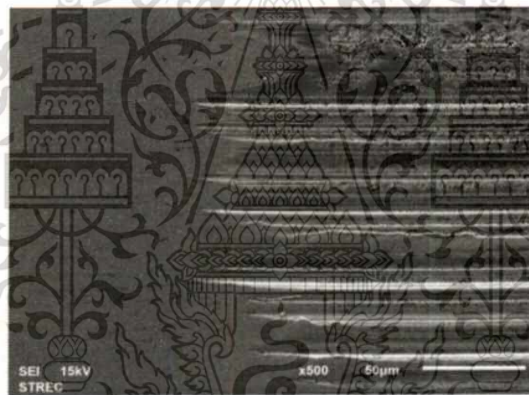


Figure 4.36 Abrasive wear on ball steel B100-2% from SEM with magnification 500 times.

The cause of abrasive wear Particles enter the clearance space between a component's two moving surfaces, bury themselves in one of the surfaces, and act like cutting tools to remove material from the opposing surface. The particle sizes causing the most damage are those equal to and slightly larger than the clearance space.

4.5.4.2 Fatigue wear

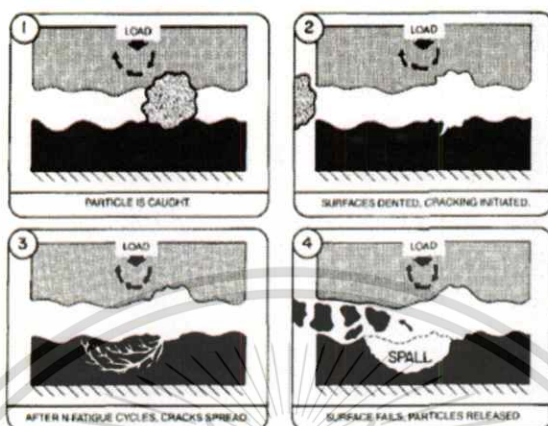


Figure 4.37 Mechanism of fatigue wear.



Figure 4.38 Fatigue wear on ball steel B100-2% from SEM with magnificent 1,500 times.

Main cause of fatigue wear fatigue wear of a material is caused by a cycling loading during friction. Fatigue occurs if the applied load is higher than the fatigue strength of the material. Fatigue cracks start at the material surface and spread to the subsurface regions. The cracks may connect to each other resulting in separation and delamination of the material pieces. Figure 4.38 the black spall represent fatigue wear of the ball steel.

CHAPTER 5

CONCLUSIONS

This research has been performed by collecting the used lubricating oil and used filters in normally operated engines. The first part study has been done based on physical and chemical properties of obtained lubricants. However, the driving manner is not included, which also effects on engine wear. The physical and chemical properties of the lubricant have quite significant relation to cause wear to the internal combustion engine. After completion this experimental analysis, the following conclusion can be drawn.

- 5.1.** The wear condition results obtained during this experiment shows; iron, copper and aluminium are the major wear components in comparison to other components. This may be due to most of dynamic components are made of iron, copper and aluminium.
- 5.2.** The data from oil condition analysis shows, degradation of viscosity is the major issue for all tested sample with compared to other properties and it degrades with longer use of lubricant. However, oxidation value not in similar pattern with increased engine age and oil age because its completed chemical features.
- 5.3.** The analysis of contamination in lubricant has been made to compare presence of soot in both lubricant groups. The result showed that higher percentage of soot with longer use of lubricant. It illustrates that the soot produced in diesel combustion is washed away with lubricant causing degradation to the lubricating efficiency.
- 5.4.** From used lubricating oil the chemical and physical properties decrease when the mileage and lubricating oil age increase such as the increasing of oxidation rate and particle of wear debris.
- 5.5.** From used oil there are in the range of 0.1 - 23 μm of particle the found in used oil by around 0.1-1 μm is soot and the size more than around 1 μm is metal wear debris.

- 5.6. From oil filter there are in the range of 3-50 μm of particle the found in SEM image by round 3 μm is soot and the size more than around 10 μm is metal wear debris.
- 5.7. From laser distribution technique there are in the range of 0.1- 100 μm by there are 2 peak of result the first peak is soot in the range 0.1 - 1 μm and the second peak is metal wear debris in the range 1 - 100 μm .
- 5.8. If we can completely make size of oil filter within 0.1 - 10 μm that effect to engine wears will be reducing.

The second part is the result from four ball tribotester, 3D optical microscope and images from scanning electron microscope by investigated of biodiesel fuel contamination by blend with new lubricating oil SAE 0w-30. After completion this experimental analysis, the following conclusion can be drawn.

- 5.9. Fuel contamination of diesel fuel and biodiesel affect to friction and wear scars of the ball by the highest wear scar and friction whereas biodiesel 100-2% and minimum friction and wear scar whereas pure lubricating oil SAE 0w-30.
- 5.10. Palm biodiesel B20-2% contamination shows the positive effects on wear and friction in which lubricity increases when compare with B7-2%, B50-2% and B100-2%.
- 5.11. The oxygen in biodiesel affects to increase wear and friction, by in this experiment whereas B100 has not only highest percent of oxygen but also friction and wear scar.
- 5.12. The four ball is the method to evaluate the sample oil lubricity, associated with the boundary lubrication. The result show biodiesel B20-2% improves the lubricity of oil contamination than B7-2% B50-2% and B100-2%, because it has viscosity than B7-2% on the other hand it has less percent of oxygen than B50-2% and B100-2%.

REFERENCE

- [1] Michalski J, Marszalek J, Kubiak K: An experimental study of diesel engine cam and follower wear with particular reference to the properties of the materials: *Wear*: 240, p.168-79(2000).
- [2] Gara L, Zou Q, Sangeorzan BP, Barber GC, McCormick HE, Mekari MH: Wear measurement of the cylinder liner of a single cylinder diesel engine using a replication method: *Wear*, 268, p.558-64 (2010).
- [3] L.Z. Pillon, *Surface Activity of Petroleum Derived Lubricants*, CRC Press, Boca Raton, FL (2010).
- [4] S. Boyde, Green lubricants – Environmental benefits and impacts of lubrication, *Green Chem.* 4, 293–307 (2002).
- [5] S. Lawford, Polyalkylene glycols, in *Synthetics, Mineral Oils and Bio-based Lubricants* (L.R. Rudnick, ed.), CRC Press, Boca Raton, FL (2009), pp. 119–38.
- [6] A. Pettersson, High-performance base fluids for environmentally adapted lubricants, *Tribol. Intl.* 40, 638–645, (2007)
- [7] US Army Corps of Engineers, Engineering and Design, *Lubricants and Hydraulic Fluids*, Engineer Manual, 1110-2-1424, Washington, DC (1999).
- [8] M.J. Neale, *Lubrication and Reliability Handbook*, Newnes, Oxford (2000).
- [9] E.W. Dean and G.B.H. Davis, Viscosity variations of oils with temperature, *Chem. Met. Engng.* 36, 618–19 (1929).
- [10] S. Verdier, J.A.P. Coutinho, A.M.S. Silva, O.F. Alkilde and J.A. Hansen, A critical approach to viscosity index, *Fuel* 88 (2009) 2199–206.
- [11] Th. Mang and W. Dresel (eds), *Lubricants and Lubrication*, 2nd edn, WileyVCH, Weinheim (2007).
- [12] W. Soetaert and E. Vandamme (eds), *Biofuels*, John Wiley & Sons, Ltd, Chichester (2009).

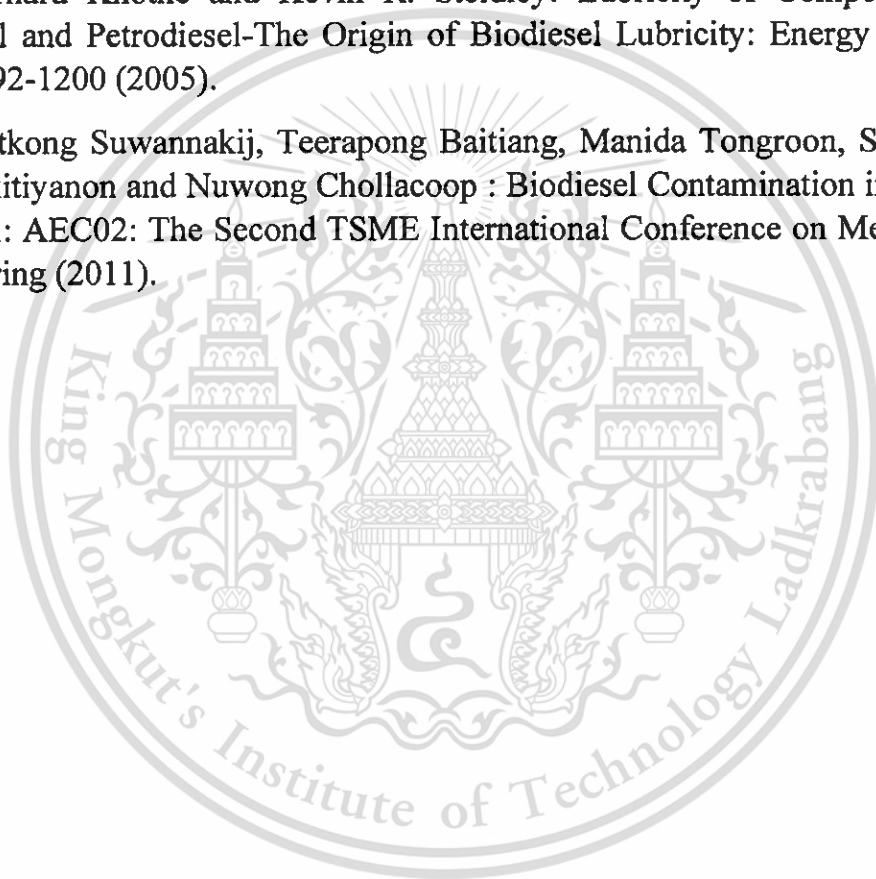
- [13] Th.F. McGowan, M.L. Brown, W.S. Bulpitt and J.L. Walsh Jr. (eds), *Biomass and Alternate Fuel Systems: An Engineering and Economic Guide*, John Wiley & Sons, Inc., Hoboken, NJ (2009).
- [14] C.V. Stevens and R. Verhé (eds), *Renewable Bioresources: Scope and Modification for Non-food Applications*, John Wiley & Sons, Ltd, Chichester (2004).
- [15] L.R. Rudnick (ed.), *Synthetics, Mineral Oils, and Bio-based Lubricants: Chemistry and Technology*, CRC Press/Taylor & Francis, Boca Raton, FL/New York, NY (2006).
- [16] James A. Addison: *Diesel Engine Lubricant Contamination and Wear*: p 1-12 Ny 11542.
- [17] Chokun Supanamok, Preechar Karin, Chi-na Benyajati, Katsunori Hanamura: *Impact of Soot in Engine Lubricating Oil on Metal Wear using Four-Ball Testing*: JSAE Paper Number: 20155318, Issued No.71-15(2015).
- [18] A.Imran*, H.H. Masjuki, M.A. Kalam, M. Varman, M. Hasmelidin, K.A.H. Al Mahmud, S.A. Shahir, M. Habibullah: *Study of Friction and Wear Characteristic of Jatropha Oil Blended Lube Oil*: ScienceDirect: *Procedia Engineering* 68, 178 – 185 (2013).
- [19] M. Shahabuddin*, H.H. Masjuki, M.A. Kalam: *Experimental investigation into tribological characteristics of biolubricant formulated from Jatropha oil*: ELSEVIER: *Procedia Engineering* 56, 597 – 606 (2013).
- [20] Lihui Dong, Gequn Shu, Xingyu Liang: *Effect of lubricating oil on the particle size distribution and total number concentration in a diesel engine*: *Fuel Processing Technology* 109, p.78–83(2013).
- [21] Mridul Gautam, Karthik Chitoor , Murali Durbha, Jerry C. Summers: *Effect of diesel soot contaminated oil on engine wear investigation of novel oil formulations*: *Tribology International*, 32, p.687–699 (1999).
- [22] J. Zhu, K.O. Lee, A.Yozgatligil, M.Y. Choi: *Effects of Engine Operating Conditions on morphology, Microstructure, and fractal geometry of Light-Duty Diesel Engine Particulates*: *Proceedings of the Combustion Institute*, 30, p.2781-2789 (2005).

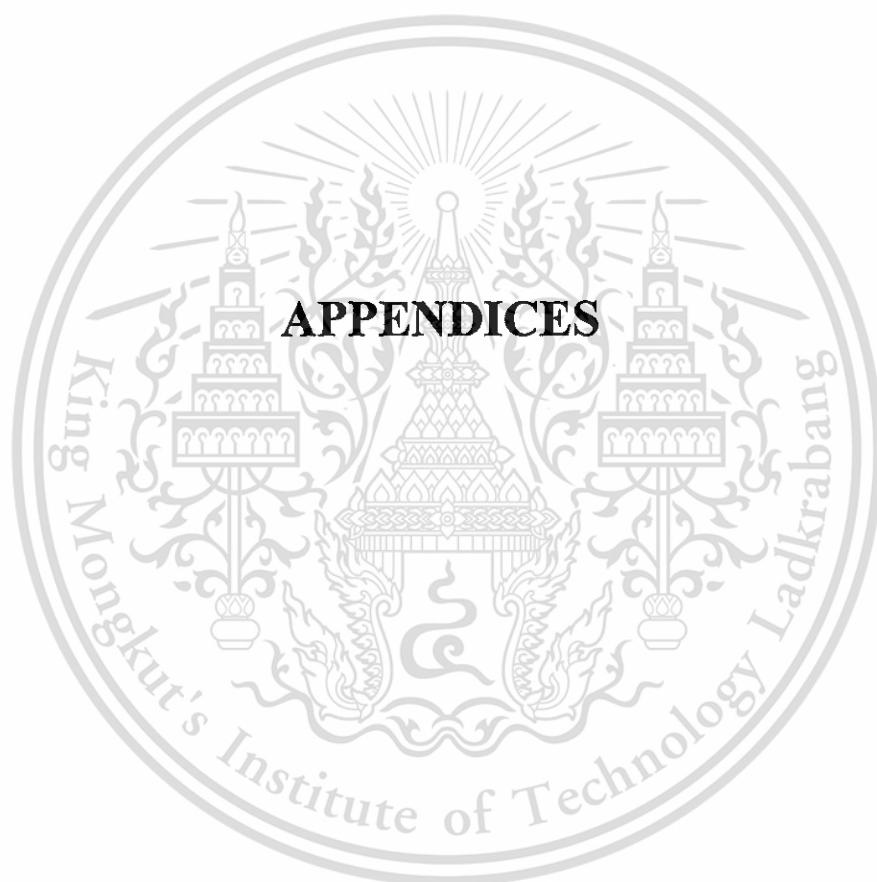
[23] M.A. Faza, A.S.M.A. Haseeb, H.H. Masjuki: Investigation of friction and wear characteristics of palm biodiesel: *Energy Conversion and Management*, 67, p.251–256 (2013).

[24] Hengzhou Wo, Karl D. Dearn, Ruhong Song, Enzhu Hu, Yufu Xu, Xianguo Hu: Morphology, composition, and structure of carbon deposits from diesel and biomass oil/diesel blends on a pintle-type fuel injector nozzle: *Tribological International*, 91, p.189-196 (2015).

[25] Gerhard Knothe and Kevin R. Steidley: Lubricity of Components of Biodiesel and Petrodiesel-The Origin of Biodiesel Lubricity: *Energy & Fuels* 19, p.1192-1200 (2005).

[26] Kiatkong Suwannakij, Teerapong Baitiang, Manida Tongroon, Sathaporn Chuhakitiyanon and Nuwong Chollacoop : Biodiesel Contamination in Engine Lube Oil: AEC02: The Second TSME International Conference on Mechanical Engineering (2011).





This material is reserved for educational use only, not allowed for commercial use.

Forbidden to modify the content, and cite the document when use.

Appendix A: Publications

5th International Conference on Mechanical Engineering in Chiang Mai, Thailand
 December 17-19th 2015

December 2014 to June 19 December 2014, Department of Mechanical Engineering, The Faculty of Engineering, Mahidol University joined the international conference on mechanical engineering or TSME ICoME 2014 in The Engineer's House, Chiang Mai and received the flag for hosting next international conference in 2015. The TSME ICoME 2015 will be held from 18-19 December 2015 at The Rajabhat Amn Charoen Prathet, which is under the project of Earmohand Engineering Technology.

Bangkok Post
KINETICS
SAMSUNG

RAIST RAYONG INSTITUTE OF SCIENCE AND TECHNOLOGY FOUNDATION

TEK **BLI** **KISTLER**
 measure. analyze. innovate.

SIAM-INTER SCIENCE-ENGINEERING CO., LTD. **KITO** **WI** **GAMMACO**

WHAT'S NEW
 TSME ICoME 2015
 155 likes

This material is reserved for educational use only, not allowed for commercial use.

Forbidden to modify the content, and cite the document when use.



AEC007

An Investigation on Physical and Chemical Properties of Lubricant Used in Diesel Engine

Phirarat Khamsriuk^{1*}, Preechar Karin¹, Kobsak Sripapha², and Hidenori Kosaka³

¹ International College, King Mongkut's Institute of Technology Ladkrabang, Bangkok, 10520, Thailand

² National Electronics and Computer Technology Center (NECTEC), Pathumthani, 12120, Thailand

³ Departments of Mechanical and Control Engineering, Tokyo Institute of Technology, Japan

*Corresponding Author: pphirarat@hotmail.com

Abstract

Lubrication is one of the most important parameter of internal combustion engine which plays a vital role to prevent engine deterioration. This research investigated physical and chemical properties of lubricants used in diesel engines. The objective of this research is to measure and compare the condition of used lubricants based on its properties. In this work, several samples of used lubricants have been taken from two different engine types, namely: engine A group and engine B group. All samples were chosen so wisely to vary ages of oil. In total seven samples were from engine A group and five samples from engine B group. The physical and chemical properties of all samples were measured on the basis of three categories; wear condition, oil condition and contamination. All the samples were analyzed using respective ASTM standard method. To visualize the obtained results, it has been plotted in to the graphs and charts. The result shows wear condition of the lubricants has mixed effect. Wear condition of total metal has been found to be higher when there is higher oxidation in lubricant. Similarly lowering in viscosity of lubricant has been observed with increase in engine operation time causing more wear. Moreover it is also observed that wear condition getting worsen with high engine speed. It can be noted that physical and chemical properties of lubricant are significantly interrelated to each other.

Keywords: Lubricant, Wear condition, Contamination.

1. Introduction

Diesel engines are increasingly used in passenger cars due to their high efficiency and performance in comparison with its counterpart petrol engine. Modern diesel engine uses direct injection system, which consists of a high pressure pump, pressure storage (common-rail) and a set of injectors. A gear pump is used as pre-feed for the fuel supply to the high pressure pump. These systems including gear pump allow injecting the diesel fuel, compressed up to 2000 bar (and even more in the future), into the combustion chamber of the diesel engine in order to increase the specific engine power. Simultaneously, fuel consumption and greenhouse gases are reduced [1]. However, a direct consequence of this high pressure is the high contact pressure and temperature at different tribological contacts involved in the diesel injection systems. In diesel injection systems, diesel fuel is not only used as combustion fuel, but also as a lubricant. Diesel fuel has become poorer in terms of anti-wear elements such as sulfur and aromatic contents. Therefore, the lubricating properties of diesel fuel have drastically decreased [2-4]. Decreasing diesel fuel lubricating properties, combined with increasing contact pressure and temperature, can lead to adhesive wear in, e.g., steel/steel tribological systems, which can – in worst cases – result in component failure. An option to solve this problem in these critical tribological contacts in diesel injection systems is the application of coatings, which reduce wear and at the same time extend the lifetime of the entire injection system [5].

Major functions of lube oil include cooling, friction reduction, and wear control. The lube oil develops lubricating film between moving surfaces, which reduces friction and wear. However, the engine oil is a depository of impurities. These are in the form of solid, liquid, and gaseous contaminants. If uncontrolled, these contaminants can build up to excessive levels. High levels of lubricant contamination cause wear of mechanical components as well as breakdown of the lube oil. The result is performance degradation, reduced engine life, and short oil service life [6-8].

In this research, the comparative study of physical and chemical properties has been done for twelve different samples taken from two engine groups. Different aged lubricant sample were chosen to see the wearing effect of the engine. The results were compared in terms of wear condition, oil condition and contamination. Where, wear condition represents the present of total metal particle like iron, aluminum, copper, chromium, oil condition evaluates the properties like viscosity, oxidation, and contamination measures presence of soot.

2. Material and Methods

In this research, twelve different types of used lubricant have been taken from two engine groups, namely group A and group B. The detail of engine specification of each engine group is shown in Table 1.

AEC007

Table 1 Engine specification

Engine parameter	A Group	B Group
1. Displacement volume	1,499 cc	2,000 cc
2. Compression ratio	14.8	16.3
3. Maximum Power	105/4000 (hp/rpm)	184/4000 (hp/rpm)
4. Maximum Torque	280/1,500-2,500 (N.m/rpm)	380/1,750-2,750
5. Engine oil	SAE 0W-30	SAE 5W-30

Seven lubricant samples were taken from group A car where five samples from group B car. Those samples were named as A1 to A7 for group A car and B1 to B5 for group B. The detail of engine age and lubricant age for each sample is shown in Table 2 below. Those lubricant samples which have equal value of car age and oil age are termed as run in condition. Here, A1, A2, A3, A4 and B1 haven't changed their engine oil since the vehicle started to driven.

Table 2 Age of tested lubricant sample

S.N	Lubricant/car sample	Engine age (km)	Lubricant age (km)
1.	A1	1,980	1,980
2.	A2	6,275	6,275
3.	A3	9,993	9,993
4.	A4	11,237	11,237
5.	A5	19,046	9,362
6.	A6	19,500	9,800
7.	A7	19,779	9,811
8.	B1	3,479	3,479
9.	B2	30,597	20,014
10.	B3	47,827	16,383
11.	B4	72,180	12,914
12.	B5	106,921	23,878

These samples were tested in standard lab to measure individual physical and chemical properties. To quantify the lubricant condition, the result has been categorized in three sub division namely; wear condition, oil condition and contamination. The standard used and the measured properties are shown in Table 3 below.

Table 3 Test properties and methods

Physical/Chemical properties	Testing standard	Unit	
Wear condition	Iron	ASTM D6595	PPM
	Aluminium		
	Copper		
	Chromium		
	Tin		
	Nickel		
Lead			
Oil condition	Viscosity	ASTM D445	cSt
	Oxidation	E2412M	Abs
Contamination	Soot	E2412M	% W

3. Result and Discussion

3.1 Wear condition analysis

Wear condition analysis covers presence of external particles in lubricating oil which primarily comes from both dynamic and static engine components during engine operation [3]. Dynamic component represents those engine components which are in motion during engine operation, e.g. piston, piston ring, fuel pump, filter, plunger, connection rod, inlet and exhaust valve etc. In other hand static components represents those engine components which do not move during operating condition of engine, e.g. fuel pump injector housing, fuel tank, fuel filter, fuel line, cylinder liner etc. In general both the dynamic and static components are metallic in nature. When the engine operates, movement of dynamic components over static component accomplish by causing wear. Those wear particles are thus washed and suspended in lubrication oil. The wear condition analysis of used lubricating oil is very useful to predict wear rate, element source, and engine condition which are always under motion with each other as well as static component [7]. Based on the analysis done in this research, the major metallic components found are discussed and compared individually in each lubrication sample.

"Fig 1" illustrates the presence of wear particle in all lubricant samples. Which shows presence of iron is predominant followed by copper and aluminium. However other particles like lead, nickel and chromium are very negligible. The reason of higher number of iron and copper particles are due to the utilization of it in manufacturing of major engine components.

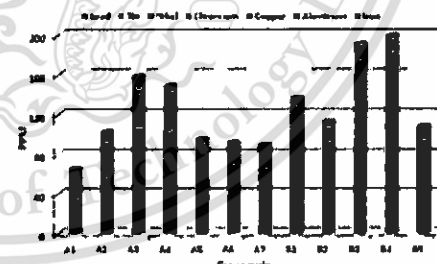


Fig. 1 Total metal wear in lubrication sample

"Fig. 2" shows total wear element for group A and group B. The comparison has been made with reference to car age and oil age respectively. The result shows higher number of wear particles for sample with shorter car age. Conversely it can be noted that longer the oil age higher will be the number of wear particle. However, two cars from group B shows high number of wear particles although the car age is longer. With the available data only, it is hard to point out reason behind higher number particles in certain sample with longer car age and shorter oil age. However, theoretically it is obvious to see higher number of wear

AEC007

particles in lubricant in case of car in run-in condition [9].

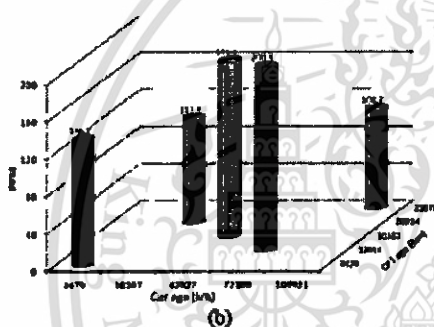
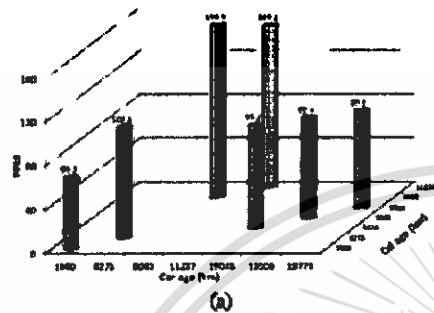


Fig. 2 Total wear metal presence in lubricant
(a) Group A, (b) Group B

3.2 Oil Condition

Oil condition analysis is a process that involves a sample of Lubricants oil, whether virgin or used. It examines oil condition, the physical and chemical properties e.g. viscosity, Total Base Number, Oxidation and Nitration value. Presence of these contaminants degrades quality of lubricant drastically. The study shows viscosity and oxidation has higher effects on lubricant. So, in this study only the viscosity and oxidation has been studied and presented. The experimental results observed in this research are presented below.

3.2.1 Viscosity

Viscosity of lubricating oil is the most important property which affects the wear rate of various engine components. The viscosity value must be within the specified range. Viscosity is inversely proportional to temperature, thus higher the temperature, the viscosity value drop causing the lubricant layer thin. Which may cause two mating part in contact ultimately causing excessive wear.

"Fig. 3" shows the viscosity of all lubricant samples comparing with its engine age vs oil age. It can be notice that, the value decreases slightly with longer use of oil. Although in spite of having large difference in oil age, there is not so much variation in

viscosity value. This may be due to the higher measurement temperature. It might be seen noticeable difference if the measuring temperature is lower.

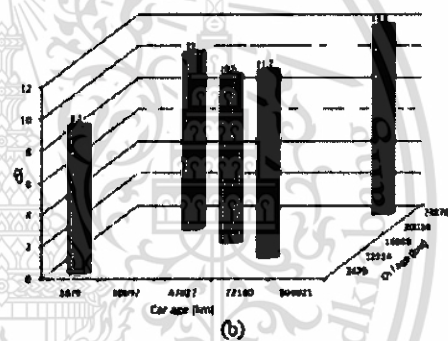
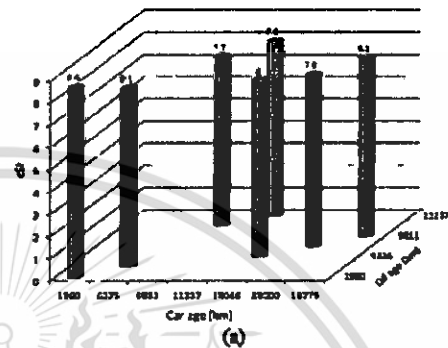


Fig. 3 Viscosity of lubricant samples
(a) Group A, (b) Group B

3.2.2 Oxidation

Oxidation is a complex primary chemical degradation process in the base oil. It can be defined as the chemical breakdown of the base oil molecules with oxygen as reagent where the principle source of oxygen is air. At lower temperatures (up to about 150 °C) the pattern of oxidation reactions may differ substantially from those at high temperatures [10].

"Fig. 4" represents the oxidation values observed for all lubricant samples. For group A it can be clearly seen that the oxidation value is getting higher with longer oil age and car age. However the trend is not clear in case of group B lubricants. As stated above, oxidation is a complex properties which is not affected only due to the longer use of lubrication, the presence of oxygen in combustion process may also plays a great role to increase its value. Moreover, higher oxidation value is not preferred for better lubrication. The increased oxidation value of lubrication may causes ionic exchange between metal components and hence increased in wear.



AEC007

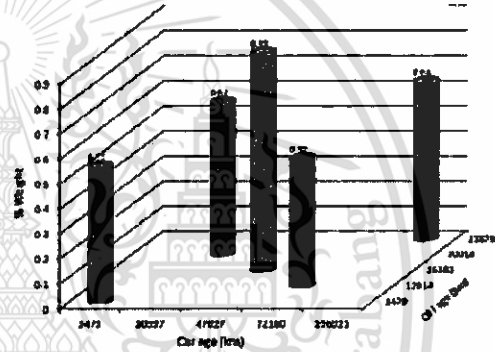
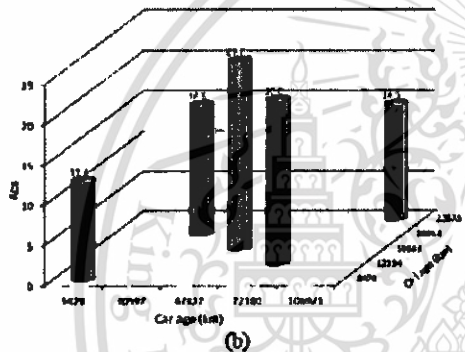
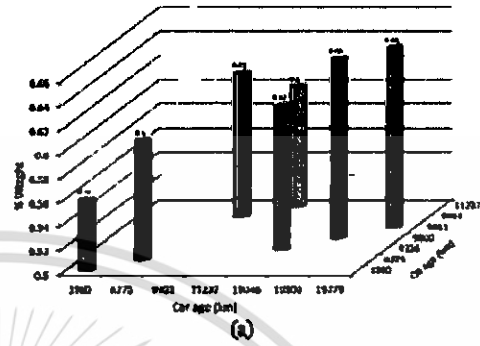
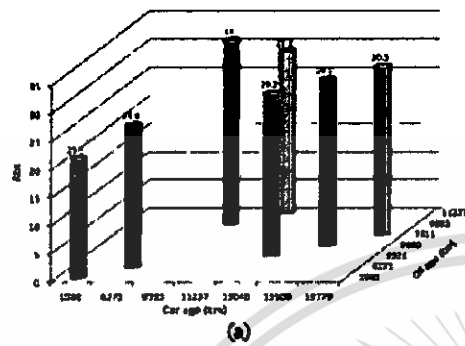


Fig. 42 Effect on Oxidation value of lubricants with engine age and oil age (a) Group A, (b) Group B

Fig. 5 Soot contamination present in lubricants (a) Group A, (b) Group B

3.3 Contamination

Diesel engine lubricant contamination is a major cause of engine component wear, leading to loss of engine performance and life. In addition, contamination accelerates breakdown of the engine lube oil, reducing its useful service life. Contaminant particles the size of or larger than the dynamic lubricant oil films separating moving component surfaces cause a major portion of diesel engine wear. The size of these harmful particles is 20 microns and smaller [11].

3.3.1 Soot

Internal combustion engines produce soot as a result of incomplete fuel combustion. Ideally, complete combustion in a cylinder would only produce carbon dioxide and water, but no engine is completely efficient. Soot occurs because of the way that fuel is injected and ignited, soot formation occurs more commonly in diesel than in gasoline engines. The fuel and air mixture in diesel engines typically do not mix as thoroughly as they do in gasoline engines. This creates fuel-dense pockets that produce soot when ignited. While the majority of soot easily escapes through the exhaust, some gets past the piston rings and ends up in the oil [12].

"Fig. 5(a)" shows the level of soot measured from all lubricant samples for group A and has been compared in terms of engine age and oil age. It can be clearly observed that the soot particles are gradually increasing with longer engine and oil age. However "Fig. 5(b)" shows the value is fluctuating with longer engine age but the soot value is noticeably higher in case of longer oil age. It is clear that, soot particles generated during combustion get washed by lubricant, which contaminates it and the value gets increased after prolong use of same engine oil.

4. Conclusion

This research has been performed by collecting the lubricant used in normally operated engines. The study has been done based on physical and chemical properties of obtained lubricants. However, the driving manner is not included, which also effects on engine wear. The physical and chemical properties of the lubricant have quite significant relation to cause wear to the internal combustion engine. After completion this experimental analysis, the following conclusion can be drawn.

1. The wear condition results obtained during this experiment shows, iron, copper and aluminium are the major wear components in

AEC007

comparison to other components. This may be due to most of dynamic components are made of iron, copper and aluminum.

2. Group B lubricants showed higher number of wear particles as compared to group A lubricants. It may be due to the difference in compression ratio, displacement volume.
3. The data from oil condition analysis shows, degradation of viscosity is the major issue for all tested sample with compared to other properties and it degrades with longer use of lubricant. However, oxidation value not in similar pattern with increased engine age and oil age because its complex chemical feature.
4. The analysis of contamination in lubricant has been made to compare presence of soot in both lubricant groups. The result showed that higher percentage of soot with longer use of lubricant. It illustrates that the soot produced in diesel combustion is washed away with lubricant causing degradation to the lubricating efficiency.

5. Acknowledgement

The author would like to thank Thailand Advanced Institute of Science and Technology and Tokyo Institute of Technology (TAIST-Tokyo Tech) for providing full scholarship and Focus lab for providing facility to test the lubricating oil.

6. References

- [1] Singh SK, Agarwal AK, Sharma M. Experimental investigations of heavy metal addition in lubricating oil and soot deposition in an EGR operated engine. *Applied Thermal Engineering* 2006;26:259-66.
- [2] Macián V, Toranzo B, Bermúdez V, Ramírez L. Assessment of the effect of low viscosity oils usage on a light duty diesel engine fuel consumption in stationary and transient conditions. *Tribology International* 2014;79:132-9.
- [3] Gora L, Zou Q, Sangcorzán BP, Barber GC, McCormick HE, Mekari MH. Wear measurement of the cylinder liner of a single cylinder diesel engine using a replication method. *Wear* 2010;268:558-64.
- [4] Uy D, Simko SJ, Carter III RO, Jensen RK, Gangopadhyay AK. Characterization of anti-wear films formed from fresh and aged engine oils. *Wear* 2007;263:1165-74.
- [5] Huang L, Bohac SV, Chernyak SM, Batterman SA. Effects of fuels, engine load and exhaust after-treatment on diesel engine SVOC emissions and development of SVOC profiles for receptor modeling. *Atmospheric Environment* 2015;102:228-38.
- [6] Michalski J, Marszałek J, Kubiak K. An experimental study of diesel engine cam and follower wear with particular reference to the properties of the materials. *Wear* 2000;240:168-79.
- [7] Vališ D, Žák L, Pokora O. Failure prediction of diesel engine based on occurrence of selected wear particles in oil. *Engineering Failure Analysis*.
- [8] Gulzar M, Masjuki HH, Kalam MA, Varman M, Rizwanul Fattah IM. Oil filter modification for biodiesel-fueled engine: A pathway to lubricant sustainability and exhaust emissions reduction. *Energy Conversion and Management* 2015;91:168-75.
- [9] Totten GE, Tung S. *Automotive Lubricants and Testing*. ASTM International; 2012.
- [10] Mobarak HM, Niza Mohamad E, Masjuki HH, Kalam MA, Al Mahamad KAH, Habubullah M, et al. The prospects of biolubricants as alternatives in automotive applications. *Renewable and Sustainable Energy Reviews* 2014;33:34-43.
- [11] Addison JA, Needelman WM. Diesel Engine Lubricant Contamination and Wear. Pall Corporation.
- [12] Supanamat C, Karin P, Benyajati C-N, Hanumera K. Impact of Soot in Engine Lubricating Oil on Metal Wear using Four-Ball Testing. *JSAE JSAE proceeding*; 2015. p. 1673-9.

 Society of Automotive Engineers of Japan

[Japanese page](#)

[Page Top](#)


[Outline](#)

[Program](#)

[Important Dates](#)

[General Information for Participants](#)

[Guidance for Speaker](#)



2016 JSAE Annual Congress(Spring)

In PACIFICO YOKOHAMA

Wednesday, May 25 to Friday, May 27, 2016

TOPICS

An Investigation on Particle Size Distribution in Used Lubricating Oils and Filters of Diesel Engine by Laser Diffraction Technique

Phiranat Khamrisruk¹⁾ Preechar Karin¹⁾ Kobsak Sriprapha²⁾ Katsunori Hanamura³⁾

1) Automotive Engineering, International College, King Mongkut's Institute of Technology Ladkrabang, Ladkrabang, Bangkok, 10520, Thailand (puaa.phiranat@hotmail.com)

2) National Electronics and Computer Technology Center (NECTEC), Thailand, National Science and Technology Development Agency (NSTDA), Klong Luang, Pathumthani, 12120, Thailand

3) Departments of Mechanical and Control Engineering, Tokyo Institute of Technology, Japan

ABSTRACT: This paper investigated the distribution of particles in used lubricating oil (SAE 0w-30) and filter in diesel engine with five samples by using laser diffraction technique. The objective of this research is to measure and compare the size distribution between used lubricating oils and filters. The laser diffraction that main technique was compared with scanning electron microscope (SEM), Ferrogram, and Filtergram to prove distribution of particle. The results of size distribution were showed in logarithm graph, in used lubricating oils the smallest particle is found to be 0.1 μm whereas largest is 23 μm . Focusing on filters, 1 μm and 100 μm are the number of smallest and largest size respectively.

KEY WORDS: Particle Size Distribution, Laser Diffraction, Lubricating Oil, Diesel Engine

1. INTRODUCTION

A Major function of lubricating oil is friction reduction, and wear control. The lubricating oil develops lubricating film between moving surfaces, which reduces friction and wear. However, the engine oil is a depository of impurities. These are in the form of solid, liquid, and gaseous contaminants. If uncontrolled, these contaminants can build up to excessive levels. High levels of lubricant contamination cause wear of mechanical components as well as breakdown of the lube oil. The result is performance degradation, reduced engine life, and short oil service life. The main cause of lubricating oil declination is contamination such as wear debris, soot particles, oxidation, water, fuel, glycol, air and heat⁽¹⁾

The way that fuel is injected and ignited, agglomerate soot formation occurs more commonly in diesel than in gasoline engines. Unlike gasoline engines where the fuel/air mixture is ignited with a spark, fuel and air entering the diesel cylinder ignite spontaneously from the high pressure in the combustion chamber. The fuel and air mixture in diesel engines typically do not mix as thoroughly as they do in gasoline engines. This creates fuel-dense pockets that produce soot when ignited. While the majority of soot easily escapes through the exhaust, some gets past the piston rings and ends up in the lubricant oil. These impacts directly on declination life of lubricating oil which contributes to wear⁽²⁾. From the literatures, we have been found several size of contaminate particle such as particle of soot, wear element, sand, dust and ect. Lihi Dong et al.⁽³⁾ investigated

Effect of lubricating oil on the particle size distribution and total number concentration in a diesel engine and the result found the peak of particle size number distribution appears at around 75 nm to 100 nm when lubricated with CI oil, while appears at around 87 nm to 115 nm when lubricated with CF oil. The concentration of the highest peak when lubricated with CF is higher than that with CI oil.

Mridul Gautam et al.⁽⁴⁾ studied Effect of diesel soot contaminated oil on engine wear investigation of novel oil formulations the result showed 0.2-0.5% of the fuel mass is emitted as small particles (approximately 0.1 nm in diameter) which consist primarily of soot. Zhu et al.⁽⁵⁾ found that the range of soot particle is 20-35 nm with a nearly normal distribution.

James A. Addison⁽⁶⁾ investigated contamination of diesel lubricant. In this test contaminant particles in different size ranges were evaluated for effect on engine wear. It was demonstrated that particles the size of the dynamic oil clearances produced the most diesel engine wear. There are various particles size that appear in the experiment since 5-100 μm by wear between piston-ring and cylinder range up to 30 μm in size and are work hardened.

M.A. Fazal et al.⁽⁷⁾ investigated friction and wear characteristics of palm biodiesel. The result sizes of the particles removed from cavities of the worn balls in B0-B50 are much bigger than 20 μm cause by adhesive wear.

Hengzhou Wo et al.⁽⁸⁾ investigated Morphology, composition, and structure of carbon deposits from diesel and

biodiesel/diesel blends on a pintle-type fuel injector nozzle the result found the carbonaceous deposition of from the emulsified biodiesel/diesel blended fuel was greater than that of diesel. The diameter of deposited carbon agglomerated particulates was approximately 10–30 μm with diesel and 50 μm with the emulsified biodiesel/diesel blend.

In this research, the comparative study of difference of particles size via used lubricant oil (SAE 0w-30) coped with filter to find out capability of trapping from filter has been done for five different samples taken from diesel engine. Different aged lubricant sample were chosen to evaluate wearing effect of the engine.

2. EXPERIMENTAL APPARATUS

2.1. Engine Specification

In this research, five different samples of used lubricating oils and filters have been taken from diesel engine, namely sample 1 to sample 5. The detail of engine specification is shown in Table 1.

Table 1 Engine Specification.

Engine parameter	Specification
Displacement volume, (cc)	1,499
Compression ratio	14.8
Maximum Power, (hp/rpm)	105/4000
Maximum Torque, (Nm/rpm)	280/1,500-2,500

2.2. Lubricating Oil Specification

SAE 0w-30 has been chosen as lubricating oil for tested diesel engine. The detail specification of used lubricating oil is elaborated in Table 2 below.

Table 2 Lubricating Oil Specification.

Properties	Specification
Viscosity @ 40 °C, (cSt)	44.5
Viscosity @ 100 °C, (cSt)	9.6
Viscosity Index	207
Oxidation, (Abs)	18.1
Nitration, (Abs)	6.1
TBN, (mg KOH/g.)	5.6

2.3. Analyzer Equipment

In this study, four different technique has been utilized to investigate distribution of particles in sample lubricating oil.

First, Scanning electron microscope (SEM), which obtains high magnification images, with a good depth of field, and can also analyse individual crystals or other features. A high-resolution SEM image can show detail down to 25 Angstroms, or better. When used in conjunction with the closely-related technique of energy dispersive X-ray microanalysis (EDX).

Next, Ferrogram provides early detection of abnormal wear of the lubricated critical internal components of mechanical systems. It analyzes the debris in system lubricants. Shows particle size, shape and color as well as quantity, identifies worn system components and to what degree, and pinpoints the cause of the wear. The cutting and normal rubbing wear was found in this paper.

Following with filtergram analysis of an oil sample can be a very useful addition to the more traditional methods of condition monitoring. The sample to be examined has to be dissolve in a solvent and filtered through a very fine membrane filter. The wear debris and other contaminants in the oil are captured on the membrane for examination under a high power microscope. The filtergram report gives an analysis of the size and composition of all the particles found as well as the wear mechanism which generated the metallic particles. Photographs of selected particles are presented on the green picture. Debris recovered from filter media can also be analyzed by this technique⁽⁹⁾.

Finally laser diffraction technique. Its used in a wide range of applications to give accurate measurement of particle size. The method is usually applicable for a particle range from 0.1 to 2,000 μm depending on the setup of the system. A particle volume fraction from 0.001 to 1.0 vol.% can normally be measured. The laser diffraction technique is based on Mie scattering developed by Gustav Mie. In order to solve for a spherical particle Mie assumed that the particle has to be isotropic, in effect no difference in which direction the incident beam strikes the particle. He also assumed that the waves of the incident light were of constant frequency, constant amplitude and parallel. The intensity of the scattered light was a function of the wavelength, λ , particle size d and the relative index of refraction n between the particle and the medium;

$$I_{\text{Scattered}} = I_{\text{Incident}} (\theta, \lambda, d, n) \quad (1)$$

When a particle is illuminated by a monochromatic source, a He-Ne laser, a diffraction pattern is created. The angle of the

diffracted light wave depends on the particle size. To solve the Mie-equation the refraction index, n , should also be known.

Fig. 1 shown below gives an overview of the different paths that can be created by a laser-light when it is hitting a droplet. When the laser beam interacts with a droplet there are four outcomes that should be considered, as shown in Fig. 1 the laser light which do not interact with the droplet can not detected. Reflection of the beam may also occur and again no droplet may be detected. The light can also hit the droplet and refracted ray could be transmitted through the droplet. The refraction process may also cause some of the light to reflect within the droplet. The relative index of refraction, n , is dependent on the fluid droplet and the surrounding gas. The most important feature of the laser diffraction technique is the light that interacts with the droplet through diffraction.

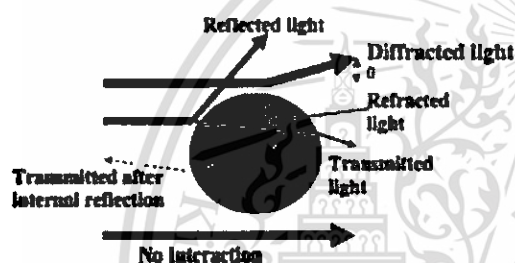


Fig. 1 Laser Light Hitting A Droplet

The diffraction angle is inversely proportional to droplet size. A detector is able to detect the angle and intensity of the diffraction pattern and thus determines the particle size.⁽¹⁹⁾

3. EXPERIMENTAL METHOD

The particle size distribution of all samples were performed with the laser diffraction granulometer and associated dispersion units. This laser diffraction particle size analyser is designed to measure particle sizes ranges from 0.02 to 2000 μm by using a blue (488.0 μm wavelength LED) and red (633.8 μm wave length He-Ne laser) light dual-wavelength, single-lens detection system. The light energy diffracted by the dilute suspension circulating through the cell is measured by 52 sensors.

The light intensity adsorbed by the material is measured as obscuration and indicates the amount of sample added to the dispersant liquid. Light scattering data are accumulated in 100 size fractions bins, which are analysed at 1000 readings per second, and compiled with Mastersizer 2000 software by using either full Mie or Fraunhofer diffraction theories. This theory is based on the assumption that; (1) particles are mineralogically

homogeneous; (2) particles are spherical; (3) the optical properties of particle and dispersion medium are known; (4) suspension dilution guarantees that light scattered by one particles is measured before being-re-scattered by other particles.⁽¹¹⁾

Five samples were taken from diesel engine. Those samples were named as Sample1 to Sample5. The detail of engine age and lubricant age for each sample is shown in Table 3 below. Those lubricant samples which have equal value of car age and oil age are termed as oil interval condition.

Table 3 Age of Tested Lubricating Oil and Filter Sample.

Lubricant/car sample	Engine age (km)	Lubricating oil age (km)
1	9,993	9,993
2	11,237	11,237
3	19,046	9,362
4	19,500	9,800
5	19,779	9,811

These samples were tested inlaser diffraction granulometer to measure individual particle size distribution. The result has been shown in logarithm graphs.

4. RESULT AND DISCUSSION

4.1. SEM/EDX

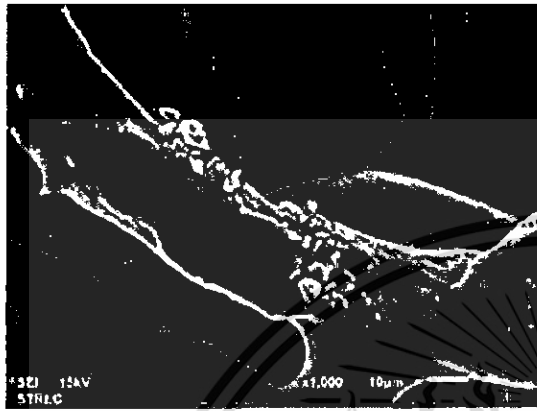
Scanning electron microscope showed wear debris, which has been measured in range of 0.5 μm to 100 μm and divided in to two main size groups. Debris diameter ranges from 0.5 μm to 5 μm , shown in Fig. 2(a), were measured using 1000X magnification scale while diameter ranges from 5 μm to 100 μm , shown in Fig 2(b), were measured with 500X magnification scale. Groups of metal wear debris were identified from energy dispersive X-ray microanalysis, that consisting O 39%, C37%, Fe 22% Ca 1.5% and K 0.37% by weight. Fig. 2 showed the largest group of wear debris, size is 80 μm the following components were present, Fe 78.08 % , O 10.66% , Al 0.92% , Cr 0.77% and Si 0.37% respectively.

From the constituent of element can observed, these group of wear debris efficacy can be inserted and read in to the fibers of filter. If a large number of wear debris metal group are found in used lubricating oil that indicates reduction in life of lubrication oil and which are very critical to cause abrasion wear in the engine. Main cause of adhesive wear occurs when the boundary layer of lubricant film between the asperities of two opposing surfaces is displaced. However other wear debris were generated by the high pressure in the combustion chamber, which is termed as combustion blow by. The wear debris by

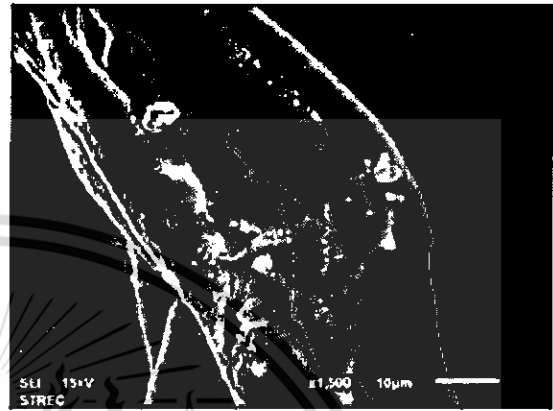
combustion blow directly affects on cylinder liner and piston rings resulting deterioration on sealing efficiency

Consequently, the deposition of soot in rings due to incomplete combustion also reduces the sealing efficiency.

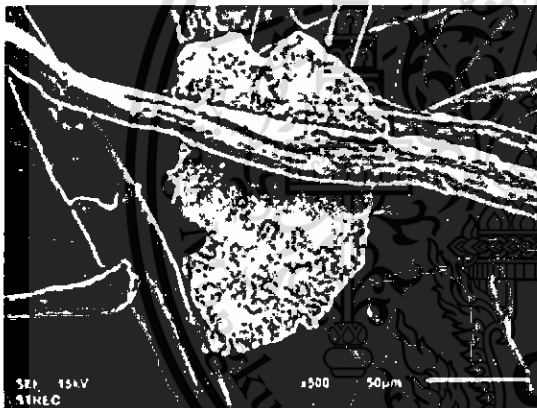
around $0.03 \mu\text{m}$ size, they often clump together to form larger particles. Individual soot particles pose little risk to engine parts, but clumps of soot can cause damage.⁽¹²⁾



(a)



(a)



(b)



(b)

Fig. 2 Small Wear Debris (a), (b) Biggest Wear Debris.

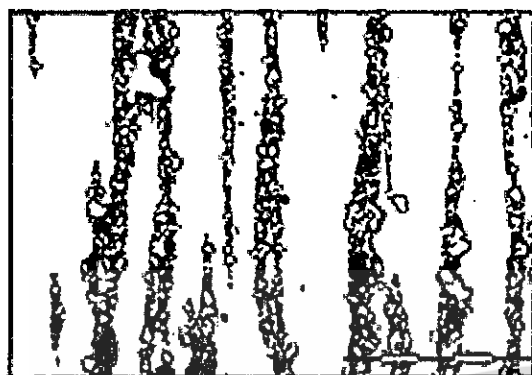
Fig. 3 Nodular Shape (a), Flat Shape of Carbon Composition.

The second group of particles pack that showed in Fig. 3(a) and 3(b) shows, particle size distribution is very wide. Particles measuring $5 \mu\text{m}$ to $20 \mu\text{m}$, identified by EDX contains two third of carbon and one third of oxygen. As noticed from Fig. 4(a), higher percentage of carbon in those nodular shapes suggests the presence of soot particles in tested lubricant. Additionally, those flat shaped particles as shown in Fig.4 (b) consists of 79.32% of carbon and 20.68% of oxygen, also seems like soot particles although the shape is different. The variation in shape may be due to the variation in carbon and oxygen percentages.

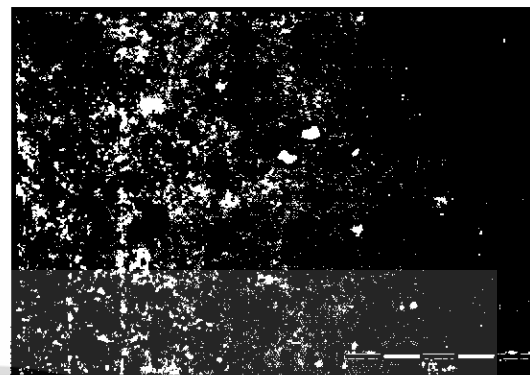
The primary soot particles contains 98 percent carbon by weight and typically spherical in shape. While most are only

4.2. Ferrogram/Filtergram

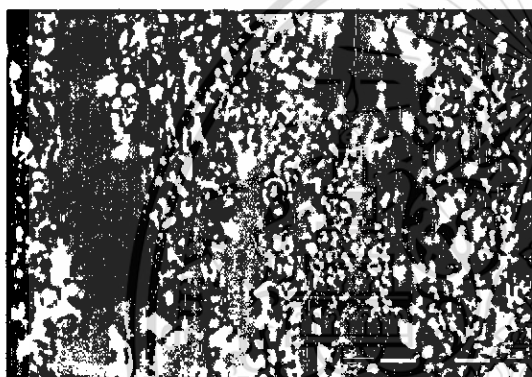
Ferrogram analysis is useful to understand the main cause of wear by evaluating debris particles found in lubricating oil. These particles consist of metallic and non-metallic matters. The metallic particle is a wear condition that separates different size and shapes of metallic dust from components like all type of bearings, gears or coupling. In Fig. 4(a) showed size of metallic wear debris, there are mainly composed of iron and have magnetic properties, $1 \mu\text{m}$ to $20 \mu\text{m}$ sizes are because of normal rubbing wear. These wear particle can be generated from general wear on the engine.



(a)



(a)



(b)



(b)

Fig. 4 Normal Rubbing Wear (a), Long curled strips (b).

From Fig. 4(b) Long curled strips of metal wear was found in the middle. These are caused by abrasion wear in lubricant system or a part of engine, misalignment or contamination of soot. If long curled strip were found considerable in used lubricating oil betoken that engine has high wear condition rate and negatively affect to the engine.

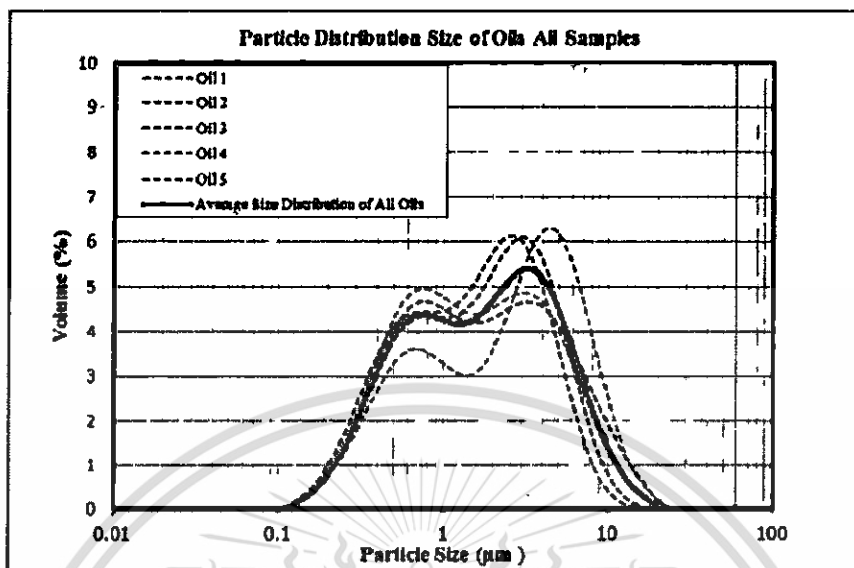
Filtergram technique is used to detected non-metallic particle which consists dirt, sand or corroded metallic particles as shown in Fig. 5. In Fig. 5(a) black points are distribution of soot appeared in grean chart, their size varies between 0.1 to 10 μm , these contamination in lubrication oil could be generated by combustion blow effect as discussed earlier.

Fig. 5(a) and 5(b) is Aluminum component wear, cause of Aluminum particles occurred were abrasion wear and misaligned with magnetic field. There are 1 μm to 10 μm Fig. 5(a) and 20 μm in 5(b) which is regarded as the normal size of wear.

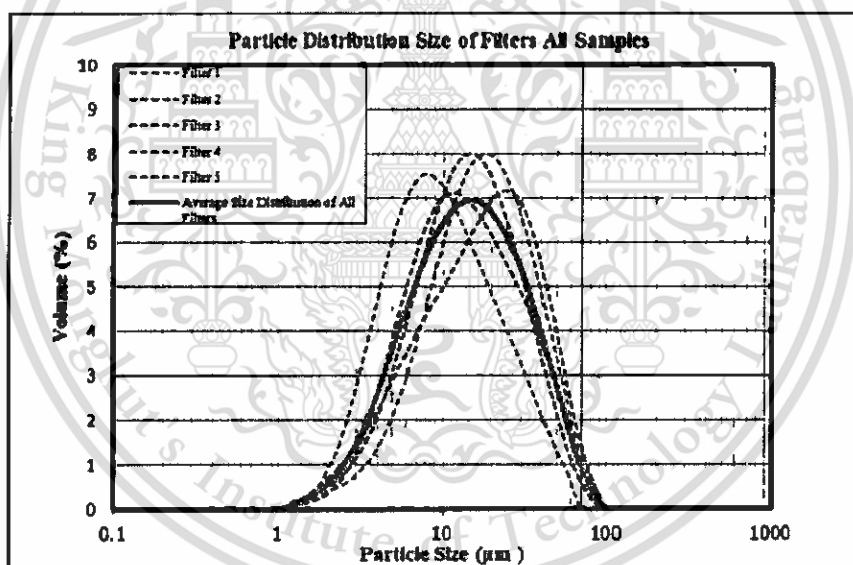
Fig. 5 Distribution of soot (a), Size of Aluminum (b).

4.3. Particle Size Distribution

To precision measure and identify the particle size distribution of lubricating oils and filters from five samples, laser diffraction technique was used for this experiment to detect size of particle. Graph.1 showed particles which appear in lubricating oils, the smallest size 0.1 μm and 23.3 μm . Blue line represent the average size of particles that existing in lubricating oils from five samples, the average of particles from lubricating oils is 2.7 μm . There are 2 peak of this blue line, the highest peak was 3.16 μm which 5.52 % and the second peak is 0.68 μm , which 4.38%. From the size distribution that showed in highest peak expected to be small wear debris group with reference to result from SEM. The second peak expected to be soot that from filtergram technique in Fig. 5 (a) or even the small particle as second peak perhaps it could be dust, sand, oxidation emulsion, water and fuel because these are many factors which contaminate within the lubricating oils.



Graph. 1 Particle Distribution Size of Lubricating Oils All Samples.

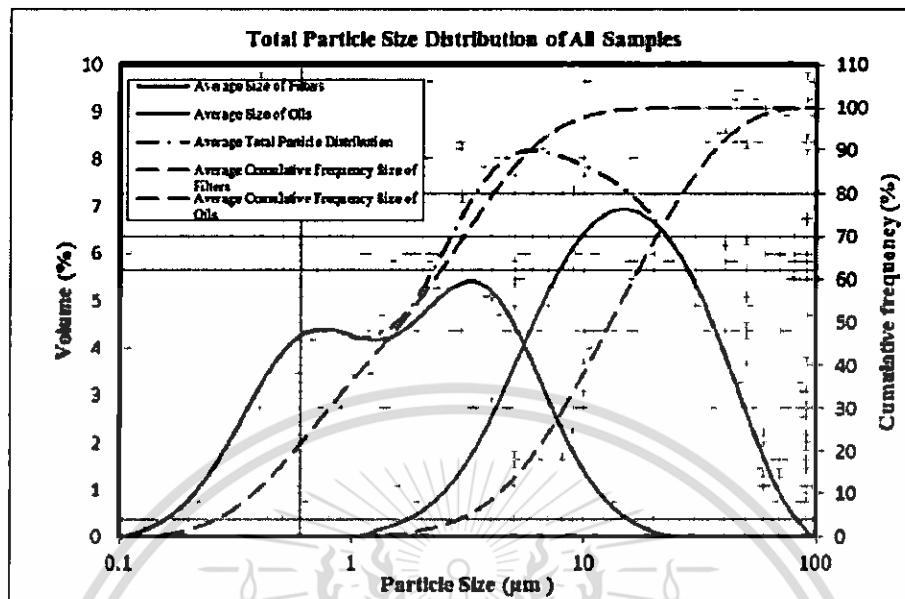


Graph. 2 Particle Distribution Size of Filters All Samples.

There are widely different in sample 4, the orange line represent particle distribution was emerged on that sample the highest peak in $4.3 \mu\text{m}$ which 6.2% and the second peak $0.68 \mu\text{m}$ size which quantity is 3.5%.

Graph. 2 showed particle distribution size of filters from five samples, from graph 1 $1 \mu\text{m}$ to $100 \mu\text{m}$ were found. Blue line represent the average size of particles from five sample is $18.54 \mu\text{m}$ the most $14.7 \mu\text{m}$ size appeared 6.94 % by volume. At the biggest size is $100 \mu\text{m}$ appeared in 0.02 % within sample 4.

From the result image from SEM can establish absolutely confident by these large particles are group of metal wear debris. Lubricating oil contamination causes wear debris which generates more contamination. This process is the chain reaction of wear. This proceeds by internal wear generating fresh wear debris; by internal wear opening dynamic sealing surfaces, allowing contaminant ingestion from the environment, and by lubricating oil breakdown generating varnishes, sludges, decrease oil life and increase worse chemical reaction with in oil.



Graph 3 Total Particle Distribution Size of Oil and Filters All Samples.

Particles size distribution were separated within 2 main parts, the first part is distribution of lubricating oil that showed in Graph. 3 red line represent the average particle distribution size of five sample their average size is 2.7 μm . Green line represents cumulative frequency of particles size of lubricating oils, show that since 0.1 μm until 1 μm size was found in 36% by volume and since 1 μm to 10 μm is 62% by volume this indicate that the mostly size of particle distribution in the lubricatings oil is 1 to 10 μm .

The second part is particle size distribution in filters, blue line represents the average particle distribution size of five sample their average size is 18.54 μm . Fink line represent cumulative frequency of particles size of filters, 1 μm to 10 μm size is 38% by volume and 10 μm to 100 μm size was found within 62% mean that mostly particle size in filter is 10 μm to 100 μm .

The black line represents total particle distribution size of all samples, that means, in the lubricant system of these five sample can generated particle size from 0.1 μm to 100 μm there are different distribution size around thousand times, by the most size of particle of this lubricant system can generate is between 4 μm to 6 μm .

The intersection of red line and blue line indicates that filter can capture particle larger than 1 μm , however filtration efficiency of filter appears better for particles larger than 4 μm . The trend of red line suggests, particles size of used lubricating oil start to decrease after 4 μm .

4. CONCLUSION

This research has been performed by collecting the lubricating oil used in normally operated engines. The study has been done based on particle size distribution of obtained used lubricating oils and filters. However, the driving manner is not included, which also effects on engine wear. The particle size distribution of the lubricant have quite significant relation to cause wear to the internal combustion engine. After completion this experimental analysis, the following conclusion can be drawn.

1. From SEM and EDX in oil filters, wear debris metal group and carbon composite was found within 0.5 μm to 100 μm and 0.5 μm to 5 μm respectively. The constitute of wear debris element can observed that these group of wear debris efficacy insert and rend in to the fibers of filter.

2. Metrallic wear debris group in ferrogram showed, they are mainly composed of iron and have magnetic properties, 1 μm to 20 μm sizes were found that is normal rubbing wear. Soot size between 0.1 to 10 μm and Aluminium 1 μm to 10 μm was detected in filtergram of filters.

3. Particle size distribution 0.1 μm to 100 μm was generated by lubricant system, from cumulative frequency that showed in green line of lubricating oils found size between 0.1 μm to 10 μm with in 98% by volume and 1 μm to 100 μm in filter. Filters can capture particle since 1 μm or more and good filtration efficiency of filter was appeared since 4 μm .

ACKNOWLEDGEMENT

The author would like to thank Thailand Advanced Institute of Science and Technology and Tokyo Institute of Technology (TAIST-Tokyo Tech) for providing full scholarship. National electronics and computer technology center (NECTEC), Thailand, national science and technology development agency (NSTDA) and Focus lab for providing facility to test the lubricating oil.

REFERENCES

- (1) Michalski J, Marszałek J, Kubiak K: An experimental study of diesel engine cam and follower wear with particular reference to the properties of the materials: *Wear*, 240,p.163-79(2000).
- (2) Gara L, Zou Q, Sangeorzan BP, Barber GC, McCormick HE, Mekari MH: Wear measurement of the cylinder liner of a single cylinder diesel engine using a replication method: *Wear* , 268,p.558-64 (2010).
- (3) Lihui Dong, Gequn Shu, Xingyu Liang : Effect of lubricating oil on the particle size distribution and total number concentration in a diesel engine : *Fuel Processing Technology* 109, p.78–83(2013).
- (4) Mridul Gautam, Karthik Chittoor , Murali Durba, Jerry C. Summers: Effect of diesel soot contaminated oil on engine wear investigation of novel oil formulations: *Tribology International*, 32, p.687–699 (1999).
- (5) J. Zhu, K.O. Lee, A.Yozgatligil, M.Y. Choi: Effects of Engine Operating Conditions on morphology, Microstructure, and fractal geometry of Light-Duty Diesel Engine Particulates: *Proceedings of the Combustion Institute*, 30, p.2781-2789 (2005).
- (6) James A. Addison: Diesel Engine Lubricant Contamination and Wear: p 1-12 Ny 11542.
- (7) M.A. Faza, A.S.M.A. Haseeb, H.H. Masjuki: Investigation of friction and wear characteristics of palm biodiesel: *Energy Conversion and Management*, 67, p.251–256 (2013).
- (8) Hengzhou Wo, Karl D. Deam, Ruhong Song, Enzhu Hu, Yufu Xu, Xianguo Hu: Morphology, composition, and structure of carbon deposits from diesel and biomass oil/diesel blends on a pintle-type fuel injector nozzle: *Tribological International*, 91, p.189-196 (2015).
- (9) Mihir Patel, CristyLeonorAzanzaRicardo, PaoloScardi, PraneshB.Aswath: Morphology, structure and chemistry of extracted diesel soot—Part I:Transmission electron microscopy, Raman spectroscopy, X-ray photoelectron spectroscopy and synchrotron X-ray diffraction study: *Tribological International*, 52, p.29-39 (2012).
- (10) A. Eddie Setekleiv, Hallvard F. Svendsen: Comparison of piezo electric particle monitor with laser diffraction technique: *Measurement*, 55, p.133-141 (2014).
- (11) F. Storti, F. Balsano: Particle size distributions by laser diffraction: sensitivity of granular matter strength to analytical operating procedures: *Solid Earth*, 1, p.25-48 (2010)
- (12) Chokun Supanamak, Preechar Karin, Chi-na Benyajati, Katsunori Hanamura: Impact of Soot in Engine Lubricating Oil on Metal Wear using Four-Ball Testing: *JSAE Paper Number: 20155318, Issued No.71-15(2015).*

AUTHOR BIOGRAPHY

Author: Mr. Phiranat Khamsrisuk
Date of Birth: 7th June 1990
Address: House No.43/1 Leang Muang 3 Lane, Leang Muang Rd, Nai Muang Sub-district, Muang District, Ubon ratchathani Province 34000, THAILAND

Education:

2009-2012: Bachelor Degree in Applied Physics,
 King Mongkut's Institute of Technology Ladkrabang.
 2013-2015: Master Degree in Automotive Engineering
 (International Program), International College, King
 Mongkut's Institute of Technology Ladkrabang.

Scholarship

2013-2015: TAIST-Tokyo Tech scholarship, the Master's Degree in Automotive Engineering, 2013-2015, founded by National Science and Technology Development Agency (NSTDA, Thailand), Tokyo Institute of Technology (Tokyo-Tech, Japan) and King Mongkut's Institute of Technology Ladkrabang (KMITL, Thailand)

2014-2015: Scholarship Energy Policy & Planning Office, Thailand, scholarship support for research

2015: DAAD Scholarship for study trip in Germany, 18-28 October 2015

Publications:

- 1) Phiranat Khamsrisuk, Preechar karin, Kobsak Sriprapha and Hidenori Kosaka, "An Investigation on Physical and Chemical Properties of Lubricant Used in Diesel Engine", The 6th TSME International

Conference on Mechanical Engineering (6th TSME - ICoME) 16th December - 18th December 2015, Regent Cha-Am Beach Resort, Petchburi, Thailand

- 2) Phiranat Khamsrisuk, Preechar karin, Kobsak Sriprapha and Katsunori Hanamura, “An Investigation on Particle Size Distribution in Used Lubricating Oil and Filters of Diesel Engine by Laser Diffraction Technique”, JSAE Annual Congress (Spring), 25th May - 27th May, 2016, Pacifico Yokohama, Yokohama, JAPAN.

