STUDY THE FRICTION PROPERTIES OF TREATED ENGINE OIL WITH AN ADDITION OF COMMERCIALIZE VS ORGANIC ADDITIVES FOR RE-USED INDUSTRIAL PURPOSE

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BACHELOR OF MECHANICAL ENGINEERING TECHNOLOGY (MAINTENANCE TECHNOLOGY) WITH HONOURS

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UNIVERSITI TEKNIKAL MALAYSIA MELAKA

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A thesis submitted
in fulfillment of the requirements for the degree of
Bachelor of Mechanical Engineering Technology (Maintnance Technology) with Honours

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2025

DECLARATION

I declare that this thesis entitled "Study the Friction Properties of Treated Engine Oil with an Addition of Commercialize Vs Organic Additives for Re-Used Industrial Purpose" is the result of my own research except as cited in the references. The thesis has not been accepted for any degree and is not concurrently submitted in candidature of any other degree.

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APPROVAL

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I dedicate this works to my beloved parents and my supervisor Dr. Muhammad Ilman Hakimi Chua Bin Abdullah, who offered unconditional love and support and have always been there for me. Thank you so much for giving me strength to finish my Final Year Project. Also to my siblings and friends who keep courage me in my hard time.

ABSTRACT

Treated engine oils with compounds like graphene and zirconia represent promising advances in lubrication technology. Graphene, a two-dimensional carbon allotrope, and zirconia, a ceramic substance, each have distinct features that can improve engine oil performance and lifetime. This abstract investigates how graphene and zirconia additions affect the friction-reducing and wear-resistant as an additives of treated engine oils. Graphene, with its extraordinary strength and lubricity, can provide a strong, low-friction boundary layer between moving parts, reducing wear and energy consumption. Zirconia nanoparticles, known for their high hardness and thermal stability, can improve the oil's wear resistance and preserve engine components at high temperatures. By adding these compounds into engine oils, manufacturers can increase the overall efficiency and dependability of internal combustion engines. In this study, ASTM D-7317 filtration and ASTM D-4172 4-Ball Tester experiment method were used. This study uses typical industrial testing procedures to examine the tribological properties of engine oils treated with varied quantities of graphene and zirconia additions. Based on the result, filtered engine oil with graphene industrial given the best result to reduce Coefficient of Friction and Wear Scar Diameter which were 0.1029µm and 0.2023mm. These values were closed to the COF and WSD of new engine oil SAE 5W-30, 0.1000µm and 0.1333mm. The next best result of additives reduced the COF and MWD were followed by zirconia 0.1056µm and 0.2033mm, next filtered engine oil with additives graphene research 0.1086µm and 0.2090 and lastly graphene technical 0.1097µm and 0.2120. These researches proved that the treated waste engine oil can be used secondary in the industry as the filtered and additives in the waste engine oil can improved in the Coefficient of Friction (COF) and Wear Scar Diameter (WSD).

ABSTRAK

Minyak enjin yang dirawat dengan sebatian seperti graphene dan zirkonia mewakili kemajuan yang menjanjikan dalam teknologi pelinciran. Graphene, alotrop karbon dua dimensi, dan zirkonia, bahan seramik, masing-masing mempunyai ciri tersendiri yang boleh meningkatkan prestasi minyak enjin dan sepanjang hayat. Abstrak ini menyiasat cara penambahan graphene dan zirkonia mempengaruhi pengurangan geseran dan tahan haus sebagai bahan tambahan minyak enjin yang dirawat. Graphene, dengan kekuatan dan pelinciran yang luar biasa, boleh memberikan lapisan sempadan yang kuat dan rendah geseran antara bahagian yang bergerak, mengurangkan haus dan penggunaan tenaga. Nanozarah zirkonia, yang terkenal dengan kekerasan tinggi dan kestabilan terma, boleh meningkatkan rintangan haus minyak dan memelihara komponen enjin pada suhu tinggi. Dengan menambahkan sebatian ini ke dalam minyak enjin, pengeluar boleh meningkatkan kecekapan keseluruhan dan kebolehpercayaan enjin pembakaran dalaman. Dalam kajian ini, kaedah eksperimen penapisan ASTM D-7317 dan ASTM D-4172 4-Ball Tester telah digunakan. Kajian ini menggunakan prosedur ujian industri biasa untuk mengkaji sifat tribologi minyak enjin yang dirawat dengan pelbagai kuantiti penambahan graphene dan zirkonia. Berdasarkan keputusan tersebut, minyak enjin yang ditapis dengan graphene industri memberikan hasil terbaik untuk mengurangkan Pekali Geseran dan Diameter Parut Pakai iaitu 0.1029µm dan 0.2023mm. Nilai ini berdekatan kepada COF dan WSD minyak enjin baharu SAE 5W-30, 0.1000µm dan 0.1333mm. Hasil terbaik seterusnya bahan tambahan mengurangkan COF dan MWD diikuti oleh zirkonia 0.1056µm dan 0.2033mm, minyak enjin ditapis seterusnya dengan bahan tambahan penyelidikan graphene 0.1086µm dan 0.2090 dan terakhir graphene teknikal 0.1097µm dan 0.2120 Penyelidikan ini membuktikan bahawa minyak enjin sisa terawat boleh digunakan secara sekunder dalam industri kerana penapisan dan bahan tambahan dalam minyak sisa enjin boleh diperbaiki dalam Pekali Geseran (COF) dan Diameter Parut Pakai (WSD).

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I want to express my gratitude to Dr Muhammad Ilman Hakimi Chua bin Abdullah, as my supervisor, for his guidance on this work. He was always there to support me and it was his drive that enabled me to finish this task with success. I express my gratitude to all the educators who supported me by giving me essential tools, without which I could not have completed this task successfully.

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LIST OF ABBBREVIATIONS

ASTM	-	American Society for Testing and Material
cm	-	centimetre
COF	-	Coefficient of Friction
DNF	-	N-dimethylformamide
f	-	Friction
g	-	gram
GN	-	Graphene
GPa	-	Gigapascal
hBN	-	hexagonal Boron Nitride
kfg	-	kilogram-force
m	-	metre
min	-	minute
mm	-	millimetre
MPa	-	Megapascal
ms	-	millisecond
MWSD	-	Mean Wear Scar Diameter
N	-	Newton
N	-	Normal force
Nm	-	Newton-metre
PAH	-	polycyclic aromatic hydrocarbons
Pa.s	-	Pascal-second
ppm	-	parts per million
rpm	-	rate per minute
SEM	-	Scanning Electron Microscopy
SOFCs	-	solid oxide fuel cells
TPa	-	Terapascal
µ	-	Coefficient
$Wm^{-1} K^{-1}$	-	watts per meter-Kelvin
WSD	-	Wear Scar Diameter
wt	-	weight percent

ZDDP	-	Zinc Dithiophosphate
ZrO_2	-	Zirconia dioxide
2D	-	Two Dimensional

CHAPTER 1

INTRODUCTION

1.1 Background

Applying a material, such oil or grease, to an engine or component to reduce friction and enable smooth movement is known as lubrication. Because they reduce friction between moving components, lubricants are essential to the operation of machinery and engines, as they increase longevity and efficiency. By forming a barrier of defense between surfaces, these materials reduce direct touch and stop deterioration. Lubricants work to limit heat generated during operation by minimizing friction, which can otherwise result in overheating and damage (Lubcheam, 2023). Lubricants also aid in sealing the spaces between components to keep dirt, dust, and other particles out of the system, which could otherwise cause problems with smooth functioning. In general, lubricants are necessary to preserve the effectiveness, dependability, and longevity of engines and machinery across a range of industries.

The most common used of lubricant was automotive industry. The global automotive lubricants market was valued at 70.2 billion U.S. dollars in 2021. The market is expected to have a compound annual growth rate of 6.3 percent between 2022 and 2030, and reach 120.1 billion U.S. dollars in the latter year (NextMC, 2023). The most popular type of automotive lubricant to find in automotive is engine oil. It is made up of base oils that have been upgraded with several additives, including detergents, dispersants, antiwear additives, and viscosity index improvers for multi-grade oils. Internal combustion engines are lubricated using motor oil.

Engine oils frequently contain additives, such as nanoparticles, to improve performance. Because of their small size, nanoparticles can enhance the viscosity, thermal conductivity, and wear resistance of lubricants. For instance, adding graphene or zirconia nanoparticles to engine oil can aid in lowering friction between moving parts, improving fuel economy and minimizing wear and tear on engine parts. Additionally, by enhancing the oil's thermal stability, these nanoparticles can let it endure higher temperatures without degrading. All things considered, adding nanoparticles to engine oil can help with durability, longevity, and improved engine performance.

1.2 Problem Statement

Petroleum-based or synthetic oil that has completed an engine's normal mileage cycle is referred to as used oil. Utilized engine oil may occasionally be refined again and utilized as raw materials for petrochemical plants or as lubricants (GreenCity, 2024). Regrettably, it is possible for used oil to accidentally or deliberately leak into the environment. By penetrating into the soil and reaching the water table, wasted engine oil can potentially leak into bodies of water. In these situations, it enters streams and waterways and eventually makes its way to lakes and rivers that are used by local communities. People's immune systems may be weakened by drinking or consuming produce that contains these substances in water. An alternative is to pour spent motor oil into sizable bodies of water, such as lakes and oceans. It consumes substances that are poisonous to marine life and have killed several species.

Engine oil that has undergone a procedure to improve performance or lengthen its useful life for a secondary application is referred to as treated engine oil. After old engine oil is extracted from an engine, it is frequently recycled or used for other purposes. Re-refining is a popular technique for preparing engine oil for subsequent use. This process entails clearing the used oil of impurities and additives in order to return it to its original state. Utilizing used engine oil as a raw material to make new lubricants or industrial goods is an additional technique. Treated engine oil can be used in various applications, such as industrial lubrication, hydraulic systems, and metalworking fluids. By treating and reusing engine oil, we can reduce the environmental impact of used oil disposal and conserve natural resources by extending the life of this valuable resource.

About 75–80% of engine oils are composed of base oil, with the remaining portion being friction modifier additives added for enhanced performance. In general, the additives serve to keep the engine clean, prevent oxidation, aid in dispersion, add detergents to the base, and increase viscosity index. Furthermore, the engine oil needs to be appropriately viscous across a wide range of operating temperatures. Two cutting-edge additions that can greatly improve engine oil performance are graphene and zirconia. Graphene, when mixed with motor oil, can increase lubricity, which lowers friction between moving parts and extends engine life and fuel efficiency. By adding a layer of protection to metal surfaces, zirconia nanoparticles can lessen wear and increase the lifespan of engine parts.

1.3 Objectives

The objectives of this project are stated as below:

1. To filter the waste engine oil and treat it as a based oil.
2. To formulate treated waste oil blended with graphene and zirconia nanoparticles.
3. To analyse the tribology properties of developed oil.

1.4 Scope of Research

The scope of this research are as follows:

1. Filtering the waste engine oil using ASTM D-7317.
2. Formulating a mixture of the graphene and zirconia nanoparticles as additives in the treated oil.
3. Testing the tribological properties using ASTM D-4172.
4. Analyzing the wear mechanism involved and using SEM.

CHAPTER 2

LITERATURE REVIEW

2.1 Introduction of Lubricant Oil

Lubricating oil is a class of refined products used to reduce friction and wear between bearing metallic surfaces. It is distinguished from other fractions of crude oil by its high (>340°C) boiling-point region (Sciendirect, 2016). The term "lubricating oil" refers to a broad category of goods that contain hundreds of different additives and basic chemicals. Although synthetic and plant-based lubricants are also utilized, crude oil distillate fractions are the most widely used type.

80-90% petroleum hydrocarbon distillate and 10-20% additives are used to give lubricating lubricants their particular characteristics. The components that make up the petroleum hydrocarbon distillate are often paraffinic or naphthenic. Table 2.1 shown properties of the paraffinic and naphthenic oils.

Table 2.1 Properties of Paraffinic and Naphthenic Oils

Paraffinic Oil	Property	Naphthenic Oil
Long carbon chains	Chemical structure	Multiple carbon rings
High	Resistance to oxidation	Medium
High	Pour point	Low
High	Viscosity	Low
Low	Volatility	High
Low	Specific gravity	High

When exposed to heat, friction, and if necessary, the exhaust gases of internal combustion engines, the unused lubricating oil undergoes changes. The quantities of polynuclear aromatic hydrocarbons in used crankcase or lubricating oil are often higher than in new oils.

There have been reports of lubricating oil leaks into the environment. The production and storage of lubricating oil have been linked to the biggest discharges. These oils, which are kept in enormous above-ground containers, have leaked extensively, much like other petroleum-based hydrocarbon products (such fuels). Underneath these tanks, the soil and groundwater may get contaminated to levels that approach saturation.

2.1.1 Lubricants in Engine Oil

One essential element in engine oil that lowers wear and friction between moving parts is lubricant. Additives and basic oils make up this mixture. The main lubrication is supplied by base oils, although additives improve performance and guard against problems including oxidation, foaming, and corrosion. Engine longevity and efficiency are increased when lubricants provide a thin coating between metal surfaces to prevent direct contact and reduce friction. Additionally, they help cool by transferring heat away from important components. Engine lifetime and smooth operation are greatly enhanced by lubricants.

An engine is a very intricate piece of equipment. Figure 2.1 below explains how oil go flows through an engine.

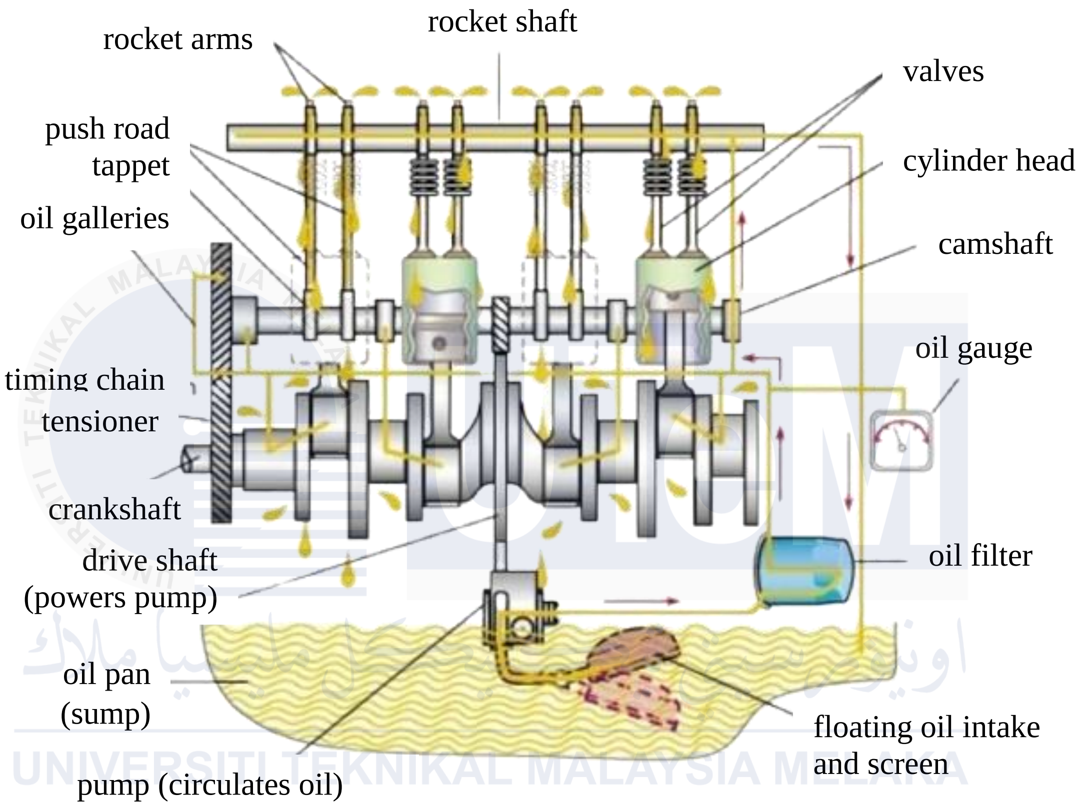

Figure 2.1 How Oil Go Through an Engine (Brittanica, 2023)

Oil is stored in the oil pan (both in the lower portion of the image), which is emptied by the oil pump. The oil is sent up to the crankshaft's primary bearings (located in the lower middle) by the pump, which transforms linear energy into rotational energy. After that, the oil passes through rod bearings, drilled oil holes in the crankshaft, and an oil line that leads to the cylinder head (in the top middle). It travels to the camshaft bearings and valves via the oil galleries. Oil thrown from the connecting rod-bearings finds its way to the pistons, rings, and pins (not seen in the illustration).

2.1.2 Important of Lubricants in Engine

Lubricants lower friction between moving parts as one of function of lubricant in engines. By creating a barrier between surfaces, they avoid wear and damage-causing direct metal-to-metal contact. Because of the decreased friction, engines run more efficiently and consume less fuel, which lowers operating temperatures. Lubricants also extend the life of crucial components by assisting in the dissipation of heat away from them (Foothills Group, 2024). Lubricants also help engines run more smoothly, quietly, and with greater overall reliability by lowering friction. In conclusion, lubricants are an integral part of any automobile system because they are necessary to preserve engine health and efficiency.

Other than that, lubricants can reduce heat-producing friction between moving parts, they are essential to engine cooling. By preventing metal-to-metal contact and forming a protective coating, they lessen wear and increase engine life. Lubricants also transfer heat from areas of friction to cooler areas of the engine, where it can dissipate more efficiently. In order to avoid overheating, which can result in engine damage and failure, this cooling effect is crucial. Maintaining ideal engine performance also benefits from proper lubrication, which minimizes energy loss from friction by ensuring that parts move smoothly. In conclusion, lubricants are crucial for engine cooling and performance in addition to being vital for minimizing wear.

Lubricants also essential to engines because they do more than merely lessen wear and friction. As cleaners, they aid in getting rid of impurities including dirt, metal shavings, and combustion by products. The longevity and performance of the engine depend on this cleaning process. Lubricants keep these dangerous materials from building up and damaging engine parts by removing them. Furthermore, lubricants have the ability to diffuse heat, which helps maintain impurities in suspension and facilitates their removal. All things considered, lubricants' ability to clean are crucial to the efficient running and extended life of engines.

2.2 Waste of Engine Oil

Engine waste oil, sometimes referred to as old engine oil or simply "waste oil," is engine oil that has been consumed and degraded to the point where it is unfit for further use. As the oil runs through the engine, it is contaminated with dirt, metal shavings, gasoline, and other substances. It is regarded as waste oil when its useful life is over and needs to be recycled or disposed of appropriately. Reusing and re-refined waste oil minimizes the demand for new oil production and has a positive influence on the environment.

There were corresponding data of available amounts of black waste of oil from industries from "Agence De L'Environmet et de la Maitise de L'Enerige" (ADEME). These wastes of oil were collected from 1999 up to 2004. Figure 2.2 below shown distribution of available amount of black waste oil many industries. Based on this data, engine waste oil was appeared to be the highest waste of black oil that have been collected in various industrial oil.

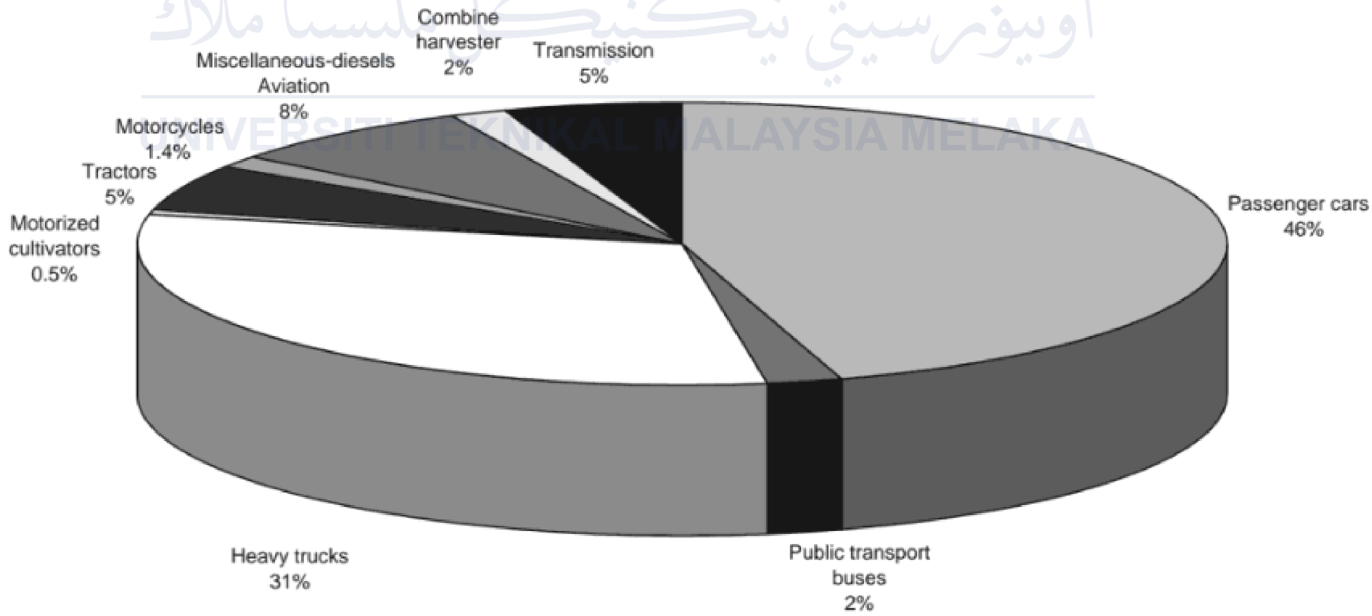

Figure 2.2 Distribution of Available Amounts of Black Waste Oil (ADEME, 2020)

2.2.1 Harmful of Waste Engine Oil

From eight major categories, waste lubricating oil has been categorized as a hazardous waste. The production process, high temperatures, and oxidation during use, combined with additives like sulphur, chlorine, and heavy metals, can result in the creation of hazardous compounds including Polycyclic Aromatic Hydrocarbons (PAH) and Polychlorinated Biphenyls (PCB). The term "waste lubricating oils" refers to a variety of spent lubricating oils used in equipment and automobiles, including waste engine oil, waste machinery oil, and waste oil from trains.

Because of its hazardous components and inappropriate disposal techniques, waste engine oil presents a serious threat to environmental degradation. Improper handling of waste engine oil can cause leaks into groundwater and soil, poisoning sources of drinking water and endangering aquatic life. The chemical makeup of the oil, which includes polycyclic aromatic hydrocarbons (PAHs) and heavy metals, can linger in the environment for a very long time and harm people, animals, and plants. These pollutants have the potential to bioaccumulate in the food chain and affect human and wildlife health. Pollution is a result of improper disposal techniques, such as pouring oil over the ground or down drains. Particulate matter and poisonous gases are among the hazardous emissions that are occasionally released into the atmosphere as a result of waste oil being burned or illegally disposed.

Other than that, he contamination of aquatic life by used motor oil can have detrimental consequences. A thin coating of waste engine oil builds up on the surface of water bodies when it is improperly disposed of or unintentionally spilled, which decreases the amount of oxygen that is exchanged between the water and air. Fish and other aquatic creatures that depend on oxygen to thrive may suffer as a result of this depleting the oxygen in the water. Furthermore, heavy metals, Polyaromatic Hydrocarbons (PAH), and other hazardous substances that may endanger aquatic life can be found in waste motor oil. These contaminants have the ability to build up in living things' tissues, which can disturb entire ecosystems and cause long-term health problems.

2.2.2 Treated Engine Oil

Used engine oil that has been treated to remove impurities and restore its lubricating qualities so it can be reused is referred to as treated engine oil. Usually, treatment consists of chemicals to neutralize acids and remove impurities and filtration to remove solid particles. Re-refining, which uses distillation and other techniques to extract the oil from impurities, is one example of an advanced treatment. Because treated engine oil may be used again in a variety of applications, including heating systems and industrial machinery, it helps conserve resources and lessens the environmental effect of disposing of spent oil.

2.3 Additives to Treat Engine Oil

Additives in treated engine oil are compounds that are mixed into the oil to improve its performance, protect the engine, and extend its life. These additives can enhance the oil's lubricating capabilities, protect against wear, minimize friction, prevent corrosion, and keep the engine clean.

Added chemicals serve a variety of functions in motor lubricants. This includes insurance for motor surfaces. Adding compounds with anti-wear, anti-rust, or anti-destructive qualities can protect motor coatings and surfaces from damage. Changing the physical properties of the oil. Thickness modifiers and pour point depressants are used to maintain the desired physical qualities (particularly consistency) over all temperatures and operating situations. This ensures a satisfactory stream and uniformity of motor oil in all conditions. Avert and manage the effects of motor shops. Added compounds with cell strengthening properties help to moderate the oxidation process, reducing the formation of unwanted motor pollutants and oil thickening (Dev Srivyas & Charoo, 2019). Based on research Pranav Dev

Srivyas, Table 2.2 shown timeline of main lubricant additives that used in the engine oil to give better performance.

Table 2.2 Timeline of Main Lubricant Additives (Dev Srivyas & Charoo, 2019)

Year	Additives Development
1930	Pour Point Depressants
1940	Antiwear Agents - ZDDP
1950	Corrosion Inhibitors
1960	Sulfonate Detergents, Salicylate Detergents, Phenate Detergents
1970	Antifoam
1980	Viscosity Modifiers, Ashless Dispersants
1990	Ashless Antioxidants
2000	Friction Modifiers
2010 and beyond	Ashless Antiwear

A multimolecular oil layer between the included surfaces maintains hydrodynamic oil lubrication. No wear occurs when surfaces do not come into touch. Nonetheless, hydrodynamic lubrication or grease isn't always possible. When loads are high or the lubrication thickness is too thin, surface ill tempers on the moving parts develop. Limit oil refers to the metal-to-metal contact between lubricant/greased surfaces. In these conditions, the anti-wear chemical bonds with the metal surface and forms a robust film to reduce grating. The adversary of wear creates low shear coatings on metal surfaces. ZDDP is by a wide margin the best multifunctional types as shown in below in Figure 2.3.

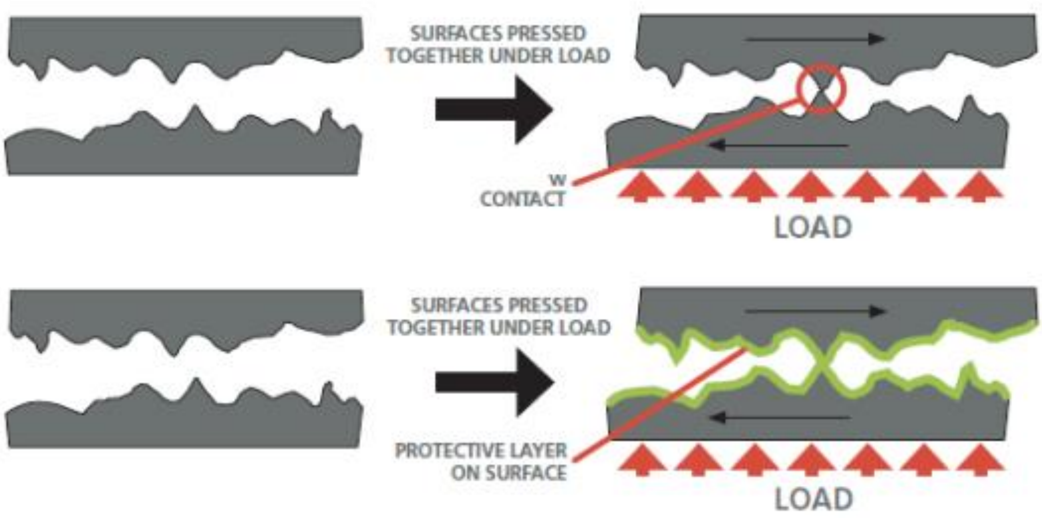

Figure 2.3 Antiwear Effect of Lubricant

2.3.1 Benefits of Additives

The use of additives in treated engine oil has numerous benefits that improve engine performance, durability, and reliability. Additives are specialized compounds that are designed to improve certain attributes of the base oil, such as friction, wear, corrosion, and thermal degradation. Here are some major advantages of adding additives in treated engine oil

Anti-wear agents and friction modifiers are two examples of additives that greatly reduce friction between moving elements in an engine. Anti-wear chemicals, such as zinc dialkyldithiophosphate (ZDDP), provide a protective coating on metal surfaces, limiting direct metal-to-metal contact and minimizing wear. Friction modifiers improve the oil's lubricity, resulting in smoother engine operation and lower energy loss.

Other than that, detergents and dispersants are essential additives that assist keep engines clean. Detergents neutralize acids and prevent deposits from forming on engine surfaces, whilst dispersants keep pollutants such as soot and sludge floating in the oil, preventing them from aggregating and producing blockages. This results in a cleaner engine interior, enabling more efficient combustion and performance.

Furthermore, anti-wear and extreme pressure additives form a protective barrier, reducing wear on important engine components like camshafts, lifters, and bearings. This results in longer engine life and fewer breakdowns. Extreme pressure additives are especially useful in high-stress situations where metal surfaces are subject to extreme frictional contact.

2.4 Graphene Nanoparticle

Graphene, a two-dimensional (2D) nano-structure made of sp2 carbons, is a component of many carbon allotropes such as charcoal, graphite, bulky balls, and carbon nanotubes. Since its discovery in 2004, graphene has been recognized as a promising nanomaterial due to its unique catalytic, optical, and electrical capabilities, as well as excellent physical features like enormous specific surface area and mechanical strength. Graphene is a sustainable, cost-effective, and accessible alternative to another nanoparticle. Usually, graphene was added to lithium grease at varying quantities, and lubrication behaviours were explored using a four-ball testing method under a variety of operational situations. Prior to the four-ball friction testing, graphene and grease materials were examined using scanning (SEM). Figure 2.4 shown schematic structure of graphene.

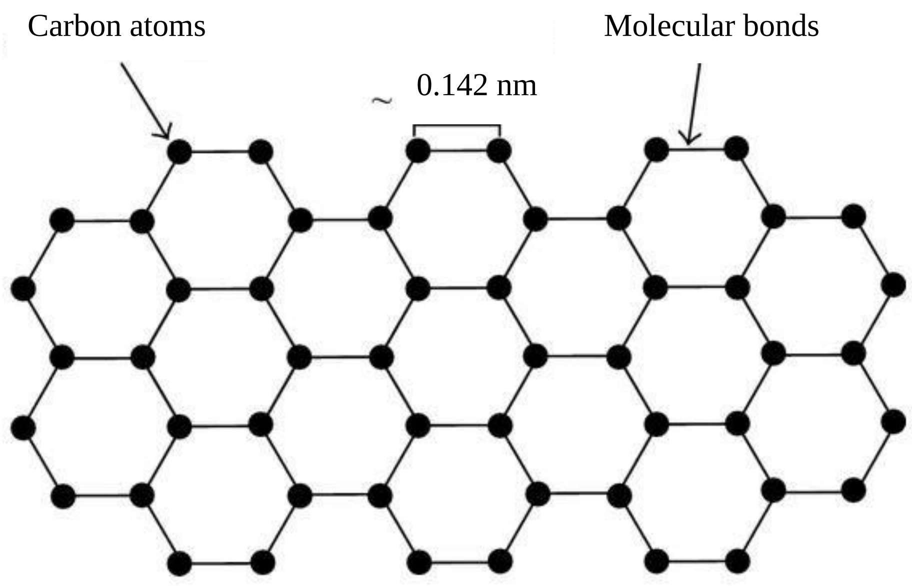

Figure 2.4 Schematic Structure of Graphene

Friction test findings show that the graphene concentration in grease varies with tribological contact circumstances to get the best lubrication behavior. Based on the findings of friction testing and worn scar morphology analysis, a lubrication mechanism was presented to better explain the interactions of grease ingredients, such as graphene, thickener, and base oil, during the shearing process. Thickener soap is thought to actively engage in the lubrication process at low speeds by releasing adequate oil into the friction contacts while under high stress.

Meanwhile, a lower graphene concentration is required to reinforce the base grease by limiting and preventing severe wear. High rotational speeds reduce the "oil-bleed" capabilities of thickeners under lower contact loads due to churning loss at high centrifugal force (Cheaptubes, 2024). Thus, additional graphene compounds are necessary to retain more oil and separate the contact surfaces. This, in turn, stimulates the production of protective tribofilm at the contact, which is critical to improving antifriction and antiwear performance. Figure 2.5 and 2.6 shown example of graphene nanoparticle lubricant additive SEM image with different graphics from research that had be done by Cheaptubes.

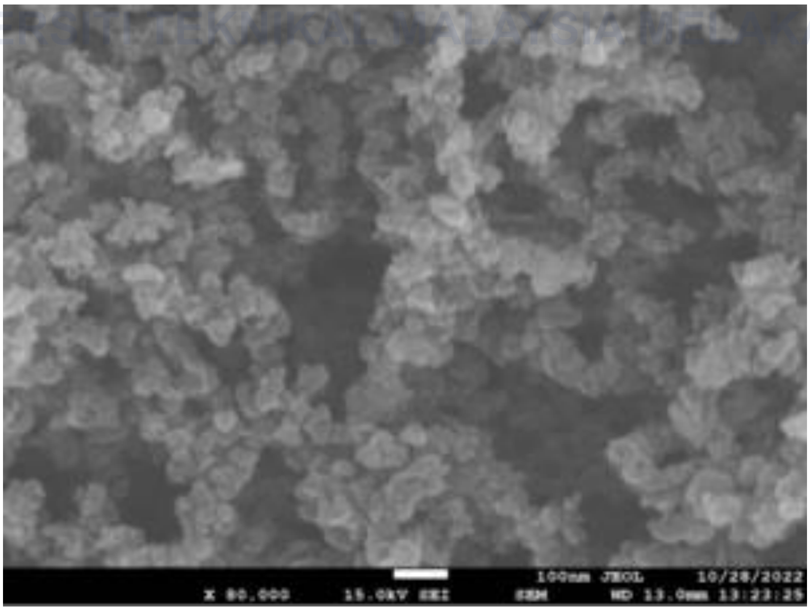

Figure 2.5 Graphene Nanoparticles Lubricant Additive SEM Image 80,000X

(Cheaptubes, 2024)

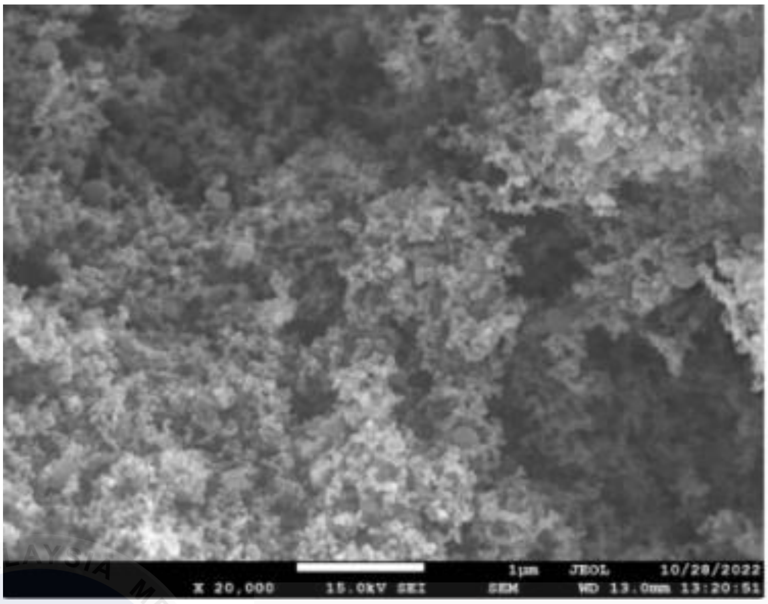

Figure 2.6 Graphene Nanoparticles Lubricant Additives SEM Image 20,000X

(Cheaptubes, 2024)

2.4.1 Properties of Engine Oil with Additives Graphene Nanoparticle

Nanomaterial-containing oils outperform traditional lubricants in terms of thermal and tribological characteristics. Nano-lubricants based on engine oil effectively minimize friction and wear, meeting or exceeding industry standards (Alqahtani et al., 2022). It was found that nanostructured additives lowered viscosity by 10-20% and improved the viscosity index. Nanodiproxamine and dehydrated carbonate sludge increased fuel viscosity at high temperatures (75 ◦C). Engine oil containing MWCNTs and zinc oxide nanoparticles performed better. More nanoparticles, together with an additive in the lubricant, increased product performance and efficiency.

Graphene's high aspect ratio and flexibility make it a great material for reducing friction and wear. Adding nanoparticles to base oil significantly improves its performance and operating features, owing to their better properties. The base oil's rheological and tribological properties were tested by Bader Alqahtani to evaluate the performance of the graphene additives. In this test, the N-dimethylformamide (DNF) was dissolved in graphene then stirred with base oil. 5 samples of these nano-oil with varying amounts of graphene ranging from 0.3% to 0.15% of the weight of the oil then tested to find different of these 5 samples in many aspects.

The pour point of base oil increases by 37% when adding graphene additives at a percentage concentration of 0.15 weight percent. However, the trend is reversed when the percentage concentration is increased to over 0.15 wt. percent, as shown in Figure 2.7.

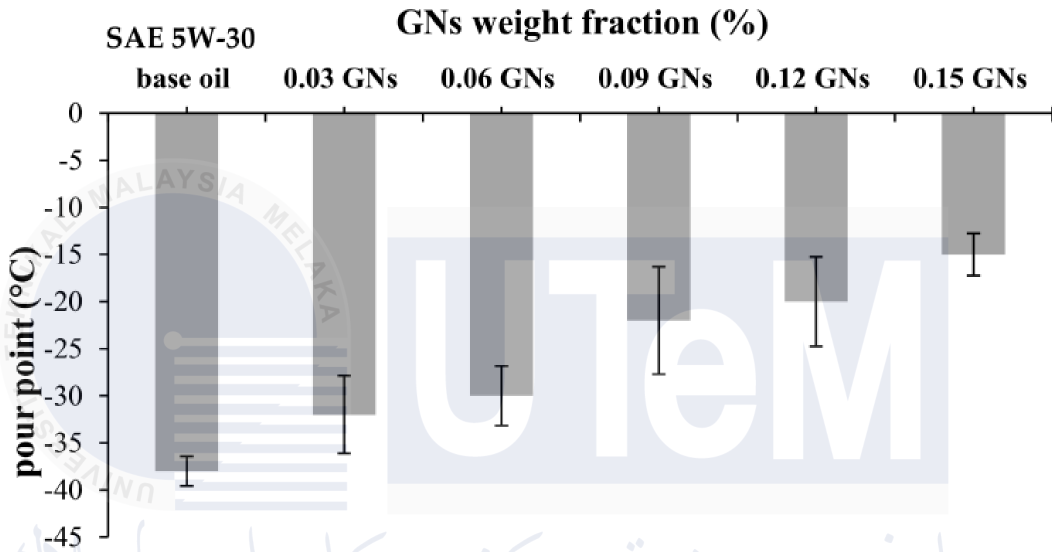

Figure 2.7 Pour Point of Base Oil and Oil with Various Percentages of Graphene

(Alqahtani et al., 2022)

The flashpoint was raised by 25.4 percent lubricants 2022, 10, 137 5 of 11 when 0.12 percent GN was added to the base oil 5W30, but when the GN concentration increased to 0.15 wt. percent, it decreased again, as shown in Figure 2.8.

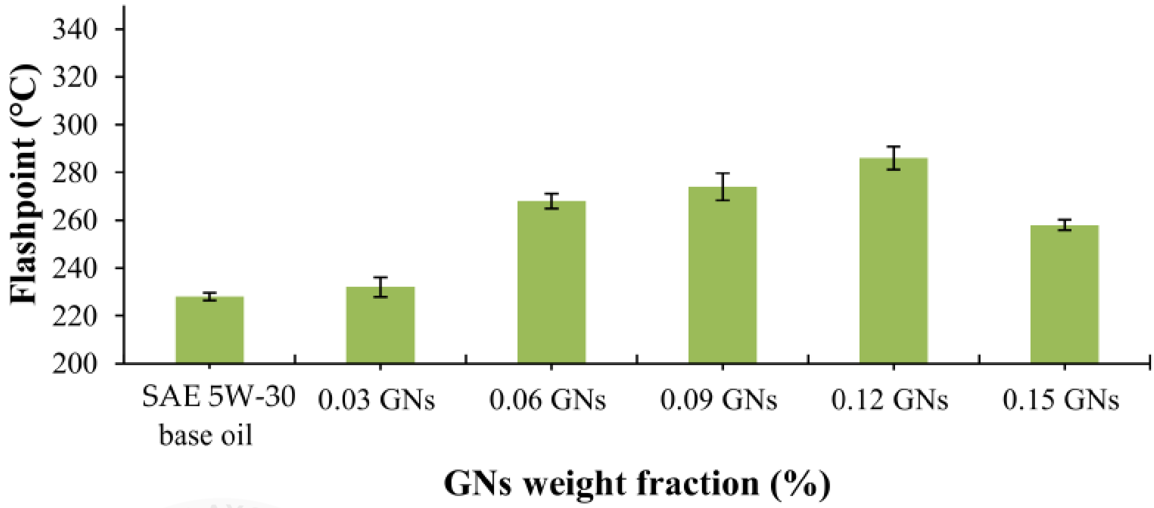

Figure 2.8 Flashpoint of Oil Alone and Oil with Various Percentages of Graphene (Alqahtani et al., 2022)

Friction or combustion generate heat flows that keep the lubricant stable in engines and machinery as they cool. Within the flashpoint limit, thermal conductivity is crucial. Graphene nanoparticles improve 5W-30 oil thermal conductivity. These materials have a high surface-to-volume ratio due to metallic oxide and nanoparticles; they improved thermal conductivity of nanofillers with a wide spectrum of uniform and stable architectures.

Figure 2.9 shows SAE 5W-30 base oil with various graphene's concentrations. Increased nanofiller volume in the graphene-containing fluid resulted in increased thermal conductivity, owing to graphene's superior thermal conductivity and good heat dispersion. At graphene proportions of 0.03, 0.06, 0.09, 0.12, and 0.15 wt. percent, thermal conductivity rises by 7%, 12%, 6%, 16.5%, 19.7% and 29.9% respectively.

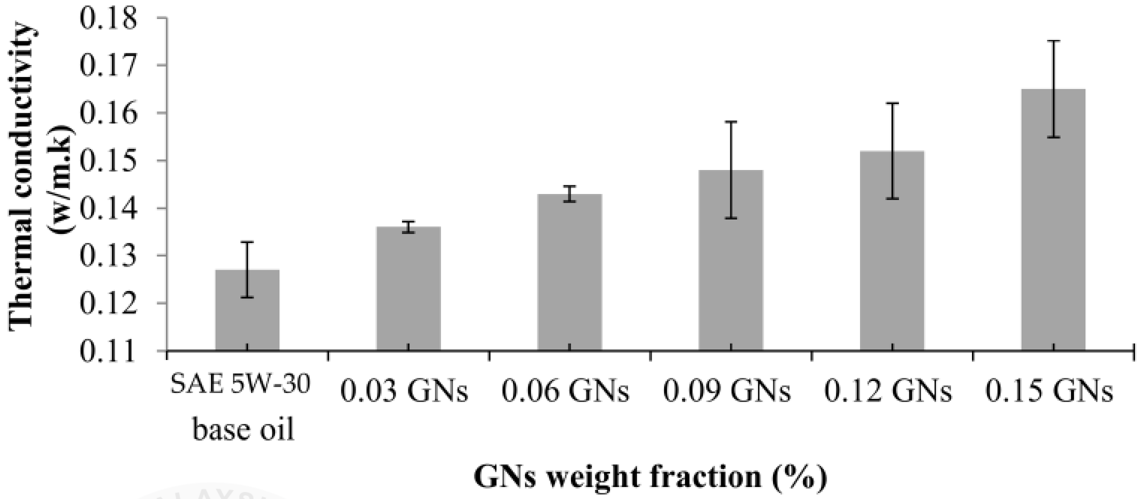

Figure 2.9 Thermal Conductivity of Different Compositions of The Nano-Oil

(Alqahtani et al., 2022)

2.5 Zirconia Nanoparticle

Zirconia, or zirconium dioxide (ZrO2), is a white crystalline oxide of zirconium that is widely employed in a variety of sectors due to its excellent qualities. It is well-known for its great crack resistance (fracture toughness), high melting point, and strong thermal and chemical stability. These qualities make it useful in a variety of applications.

Zirconia is utilized in the ceramics industry to make advanced ceramic components such as cutting tools, bearings, and dental implants due to its hardness and wear resistance. It is also utilized in the manufacture of ceramic coatings for thermal barriers in jet engines and gas turbines.

In the electronics industry, zirconia serves as an electrolyte material in solid oxide fuel cells (SOFCs) because of its ionic conductivity at high temperatures. This property is crucial for efficient energy conversion in SOFCs.

In the medical field, zirconia is used for dental crowns and prostheses due to its biocompatibility and aesthetic appeal, resembling natural teeth as shown in Figure 2.10.

Figure 2.10 Zirconia Use as Dentures in Medical Field.

Additionally, zirconia is utilized as a refractory material in furnaces and kilns, providing thermal insulation and structural integrity at high temperatures. Its chemical inertness also makes it suitable for use in chemical processing equipment and as a catalyst support in petrochemical applications.

2.5.1 Tribology Properties of Engine Oil with Additives Zirconia

There was test that been completed by Rajmund Kuti to investigate the tribological properties of nanoscale spherical zirconia nanoparticles as friction reduction and antiwear lubricant additives in fully formulated engine oil. Zirconia nanoparticles were homogenized in a pure Group III base oil. Friction and wear trials with 0.4 wt% zirconia demonstrated outstanding antiwear capabilities. Nanoparticle saturation of surface grooves resulted in smoother contacting surfaces (Kuti et al., 2022).

The study on the tribological characteristics of zirconia nanoparticles in Group III base oil with a kinematic viscosity of 4 cSt determined at 100 ◦C served as the foundation for this work. The current work presents the physical properties of zirconia nanoparticles that were examined in the previous paper, as shown in Figure 2.11. This publication reports that nanoparticles exhibit outstanding antiwear characteristics by filling surface grooves and creating a smoother contact surface. Larger agglomerates were also observed on the worn surface.

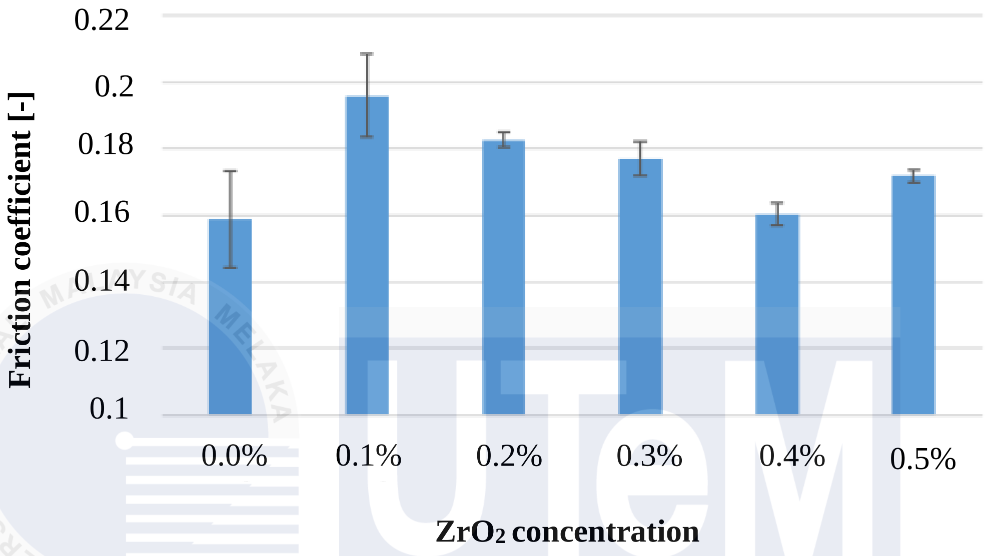

Figure 2.11 Tribological Results of Zirconia Nanoparticles in Neat Group III Type Base Oil Under 100 N Load (Kuti et al., 2022)

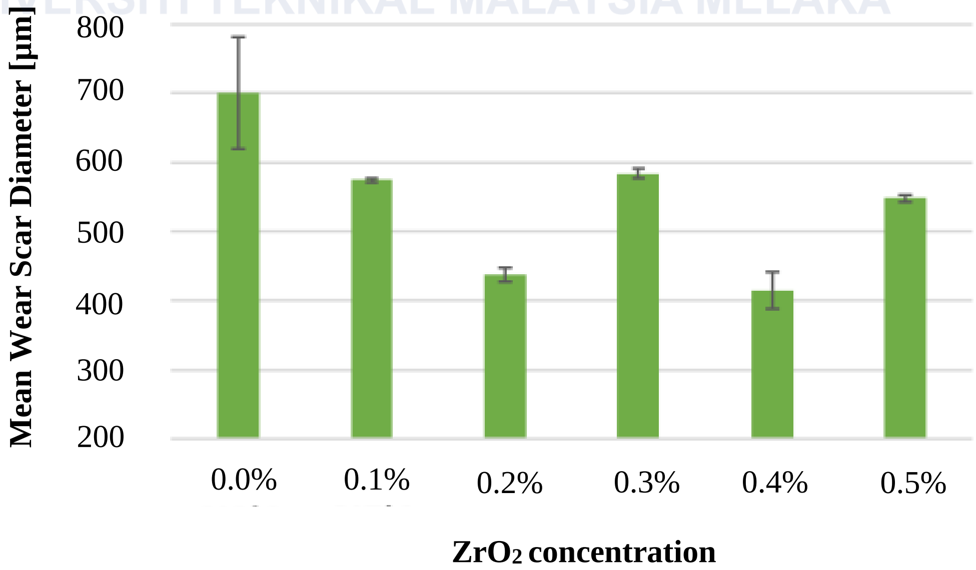

Figure 2.12 Mean Wear Scar Diameter on Ball Specimen with Different Zirconia Concentration (Kuti et al., 2022)

Figure 2.12 above shows mean wear scar diameter on the ball specimen with additives of zirconia with different concentration. The results with zirconia in base oil suggest that it can improve tribological qualities on rubbing surfaces. Further research into its potential in fully formulated lubricants is warranted.

Two engine oils were mixed with 0.5 wt% of zirconia nanoparticles. Tribological tests were performed on these samples under two distinct loads (200 N and 300 N). At least three independent tribological measurements were conducted for each variable (normal force, engine oil type, and concentration) to determine the lubricant samples' tribological properties. The results of measurements with 0.5 wt% zirconia added engine oils can be observed in Figure 2.13 and 2.14.

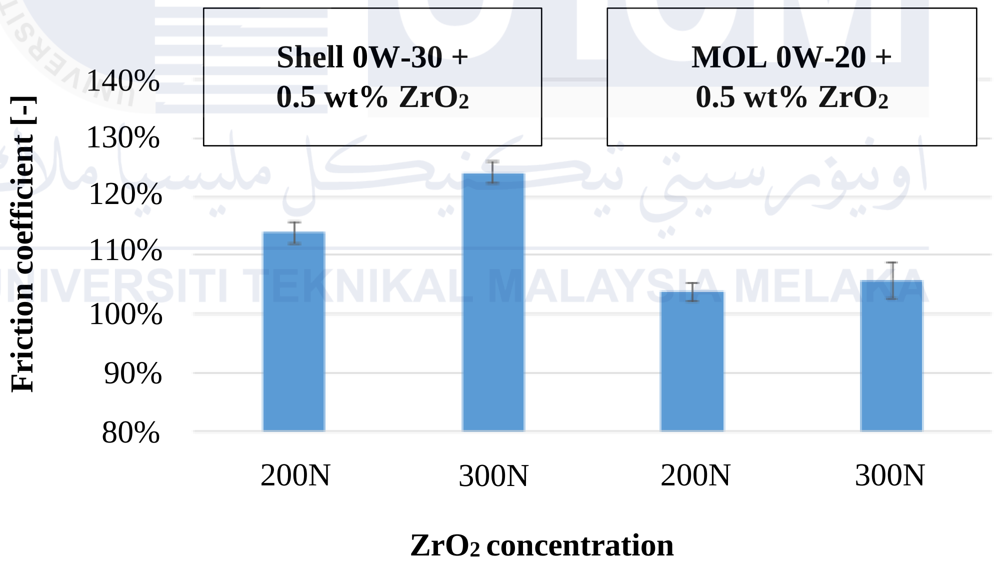

Figure 2.13 Comparison of The Tribology Result of Zirconia Nanoparticles in Two Different Formulated Engine Oil (Kuti et al., 2022)

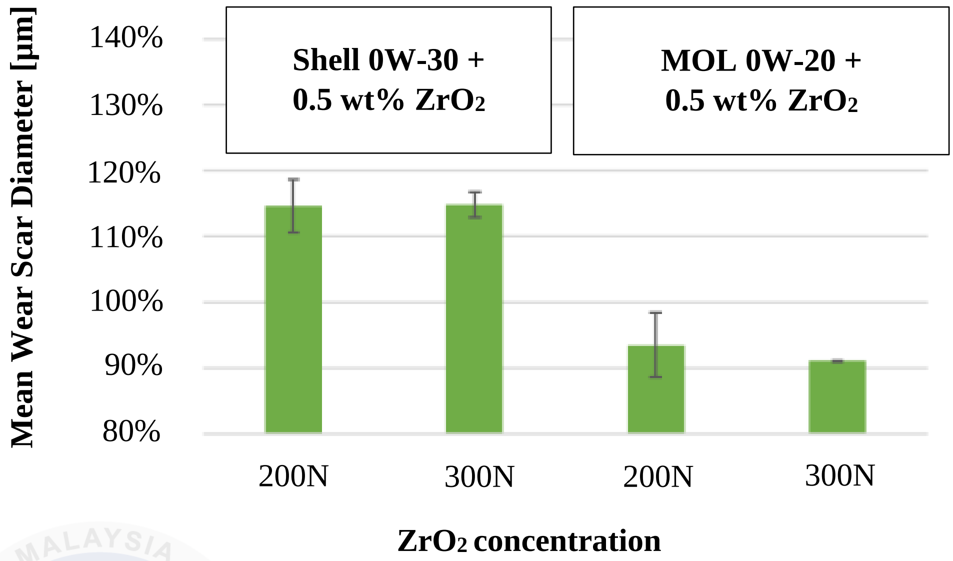

Figure 2.14 Mean Wear Scar Diameter Result on Two Different Formulated Engine Oil (Kuti et al., 2022)

The tribological data reveal a substantial difference between the two engine oils. The additional zirconia nanoparticles increased both the measured friction coefficient and mean wear scar diameter value in the Shell 0W-30 oil, whereas they provided positive antiwear properties with only a slight friction increase in the MOL 0W-20 oil with the same nanoparticle concentration.

2.6 Friction

Friction is the resistance that one surface or object faces when it passes over another. It is a force that opposes the relative motion or tendency to move two surfaces in touch. Friction is caused by microscopic interactions between surfaces, and even ostensibly smooth surfaces include flaws that generate resistance.

There are three types of friction: static friction, which keeps an object from moving when a force is applied; kinetic friction, which acts on moving objects; and rolling friction, which happens when an object rolls over a surface. Static friction is often greater than kinetic friction, which means it takes more force to start moving an object than to keep it moving. Figure 2.15 shown simple explanation of friction in diagram.

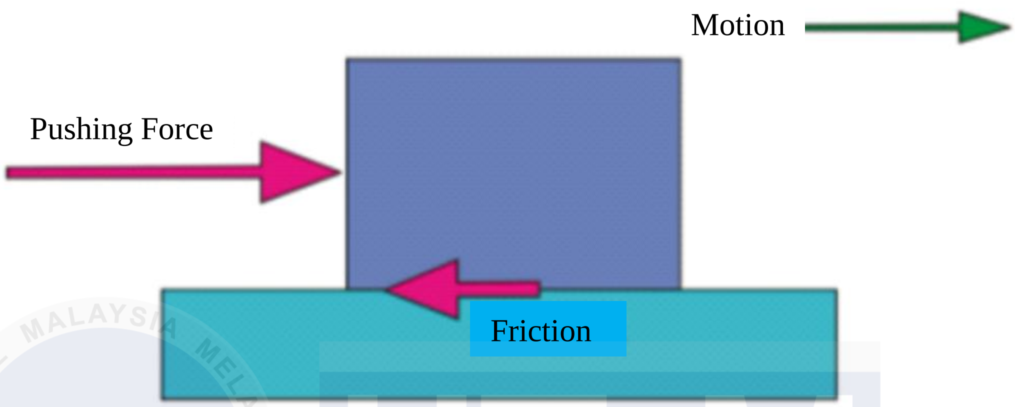

Figure 2.15 Simple Explanation of Friction

The basic equation of friction is given by:

$$f = \mu N \qquad \text{Eq 2.1}$$

Where f is friction force, μ is coefficient in friction and N is normal force.

2.6.1 Cause of Friction

Friction in engines is mostly created by the interactions of moving parts such pistons, cylinders, bearings, and camshafts. These components function at high pressures and temperatures, intensifying minuscule contact forces. Engine surfaces, despite being manufactured with high precision, are not totally smooth, it has minute flaws that grab on each other, providing resistance to motion. Furthermore, when engines run, metal surfaces expand and contract, changing the contact dynamics and adding to friction.

The engine oil's viscosity also plays an important influence. While the goal is to produce a lubricating film to decrease direct metal-to-metal contact, insufficient lubrication or oil breakdown due to high temperatures and impurities can cause greater friction. Wear particles created during normal operation can worsen friction by becoming lodged between moving parts, resulting in more abrasive contact points.

The presence of heavy loads and quick movements also contributes to the ongoing development of frictional forces. Effective engine design and the use of high-quality lubricants with appropriate additives are required to reduce friction, which improves efficiency, reduces wear, and improves the engine's overall performance and longevity.

2.6.2 Type of Wear Occur

Wear in engine refers to the slow degradation of engine components caused by friction, high temperatures, and chemical reactions. Abrasive wear is generated by impurities in the oil, while adhesive wear is caused by metal-to-metal contact. Other types of wear include corrosive wear from chemical reactions with combustion by products and fatigue wear from repetitive stress cycles. Effective lubrication, frequent maintenance, and the use of high-quality engine oils with appropriate additives can all help to reduce wear. Proactive steps such as monitoring oil condition and using filtering devices can assist extend engine life and maintain peak performance.

Friction between a hard-rough steel surface and a softer surface, such as a bearing metal, can result in abrasive wear (Khruschov, 1974). The asperities, or rough projections, on the steel surface function as cutting tools, gouging and ploughing into the softer metal. This removes material from the softer surface, resulting in wear particles and grooves. The severity of abrasive wear is influenced by elements such as material hardness differential, steel surface roughness, contact load, and the presence of abrasive particles. This type of wear can substantially shorten the life and performance of the bearing. Figure 2.16 explain how abrasive wear occur.

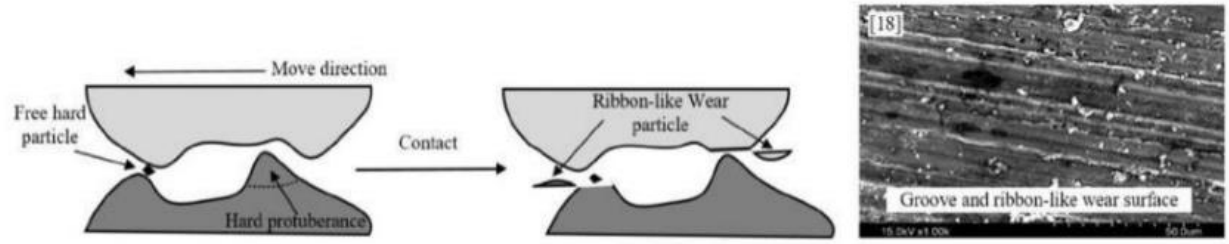

Figure 2.16 Abrasive Wear

Adhesion wear, also known as adhesive wear, occurs when two metal surfaces rubbing against each other and begin to deteriorate (Hurricks, 1973). It occurs when two metal surfaces slide against each other, causing material transfer between them. This occurs when microscopic high points (asperities) on surfaces come into touch under pressure, causing them to bind or adhere temporarily. As the surfaces move, these bonds break, removing material from one and depositing it on the other. This process causes surface damage, such as galling and scuffing, and can increase friction and wear detritus. Adhesive wear is affected by elements such as stress, surface roughness, and material compatibility. Figure 2.17 explain how adhesive wear occur.

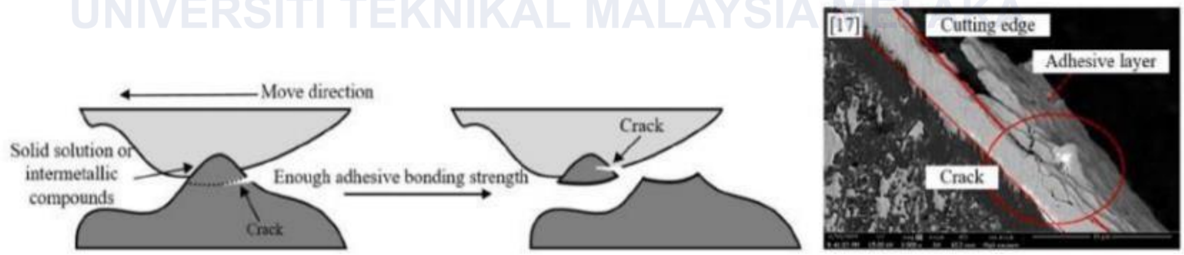

Figure 2.17 Adhesive Wear

CHAPTER 3

METHODOLOGY

3.1 Introduction

In this project, the first step was started with treat waste engine oil as a clean-based oil by filtering using ASTM D-7317. Next, the filtered engine oil was mixed with some additive composition which were graphene industrial, graphene research, graphene technical and zirconia nanoparticles and homogenizing ultrasonic tools was used in his process. 6 samples of combination will be collect as shown in Table 3.1. Then, the test ASTM D-4172 was used and analysed the result to find the wear mechanism involves used SEM. Lastly, conclude and decided the best solution of the mixture.

Table 3.1 5 Samples of Combination Treated Engine Oil and Additives

No of sample	Combination
1	New engine oil SAE 5W-30
2	Filtered engine oil + graphene industrial grade
3	Filtered engine oil + graphene research grade
4	Filtered engine oil + graphene technical grade
5	Filtered engine oil + zirconia
6	Waste engine oil SAE 5W-30

3.1.1 Flow Chart

This project involved several parts of process. This project study was taking about 2 semesters as the project process is shown in Figure 3.1. The flow chart was described all the process to analyse the results of treated engine oi lubricant by utilizing nanoparticles from the beginning until the end of the process.

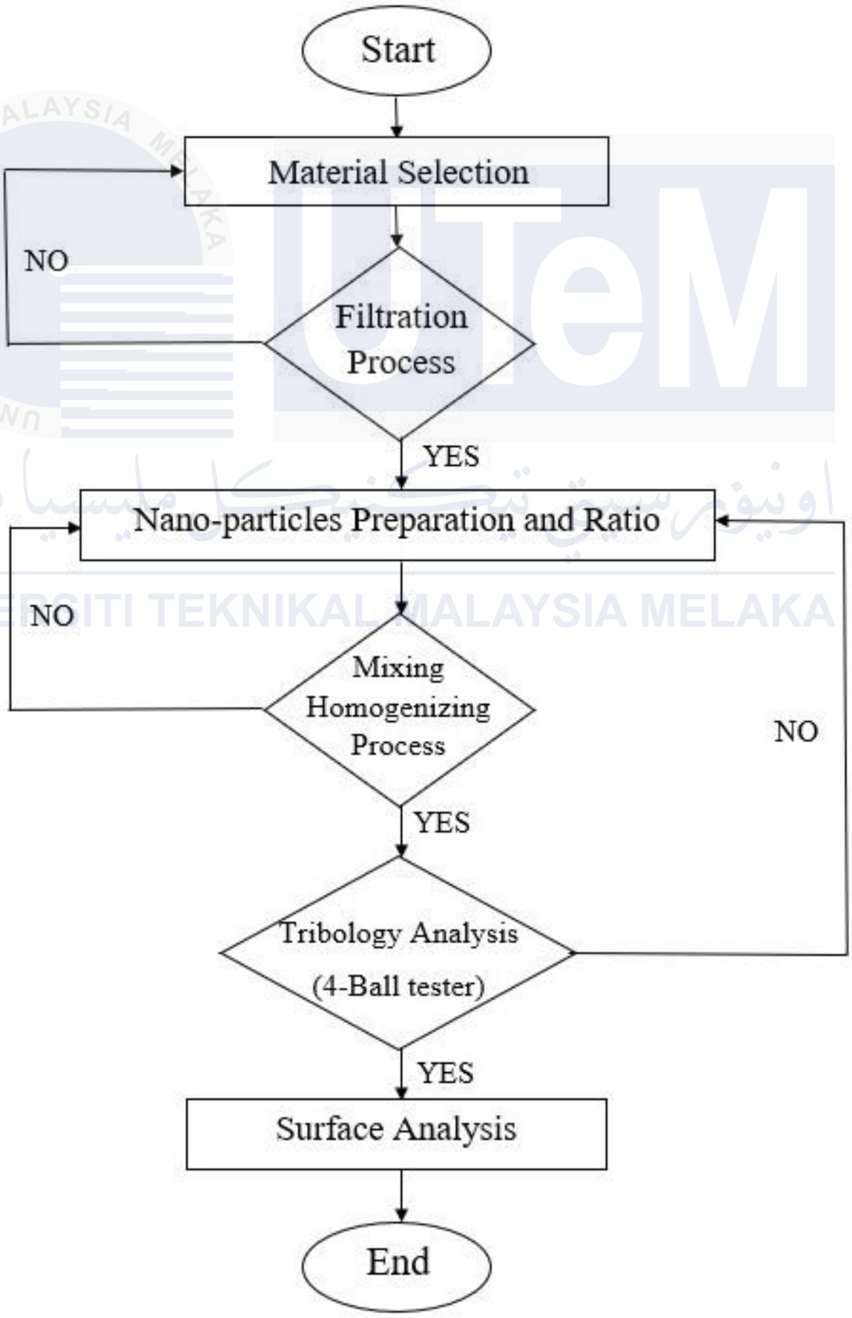

Figure 3.1 Flow Chart

3.2 Material Selection

Engine oil additive selection should take into account a number of important characteristics. Initially, the additives ought to improve the lubricating qualities of the oil, lowering wear and friction on engine components. Additionally, these additives will raise the viscosity index of the oil to guarantee steady operation at various temperatures. In order to prolong the life of the oil and safeguard engine components, additives should stop oxidation and corrosion. To keep engines clean, they should also prevent foaming and offer efficient contamination dispersal. It's also critical that the additives and components used in the engine work together. Also, consideration should be given to regulatory compliance and environmental impact.

3.2.1 Waste Engine Oil

The basic properties of waste engine oil that was used was shown in Table 3.2. Waste of synthetic oil was used since the 2019-year, waste synthetic oil has become a frequent component of most car models that use synthetic engine oil. The utilization this type of synthetic waste oil because it makes it simpler to compare the various mixture types in the final product. This waste engine oil will be bought from workshop and will be filter using ASTM D-7317.

Table 3.2 General Properties of Waste Engine Oil (Hidayah et al., 2023)

Parameter	Value
Specific Gravity	0.93
Viscosity	0.12 Pa.s
Flash Point	120°C
Iron Content	50ppm

The oil used in this project was SAE 5W 30. This multi-grade motor oil SAE 5W-30 was made to work in a range of temperatures and environments. In cold weather, the "5W" represents the oil's viscosity at low temperatures, making engine starts easier. In warmer temperatures, the "30" indicates the oil's viscosity, guaranteeing efficient lubrication while the engine is operating. Usually, this oil is a mixture of base oils plus performance-enhancing additives including dispersants, detergents and anti-wear agents. Many automakers and drivers like SAE 5W-30 because it offers dependable engine protection and performance across a broad temperature range.

3.2.2 Graphene

Graphene, a two-dimensional carbon allotrope, was increasingly used in treated engine oil for its remarkable properties. By lowering friction between moving parts and increasing fuel economy, it improves lubrication. Because of its excellent heat conductivity, graphene helps dissipate heat, lowering engine wear and prolonging oil life. Engine longevity was increased by its remarkable strength and oxidation and corrosion resistance. Graphene can also ensure optimal engine performance by increasing oil viscosity and stability over a broad temperature range. Its application to treated engine oil represents a noteworthy development in lubrication technology, provided enhanced efficiency and protection above conventional additives. Table 3.3 shows properties of graphene. There were 3 grades of graphene used in this project research.

Table 3.3 General Material Properties of Graphene (Sasmita et al., 2020)

Properties	Value
Color	Black
Electron Mobility	~2 x 10^5 cm^2 V^{-1} s^{-1}
Current Density	~10^9 A cm^{-1}

Velocity of Fermion (Electron)	~10^6 ms^{-1}
Thermal Conductivity	~5000 Wm^{-1} K^{-1}
Tensile Strength	~1.5 TPa
Breaking Strength	42 Nm^{-1}
Transparency	~97.7%
Elastic Limit	~20%
Surface Area	~2360 m^2 g^{-1}

Table 3.4 below was described 3 grades of graphene that were used in this project research.

Table 3.4 Three (3) Grades of Graphene Explanation

Graphene Grade	Description
Industrial Grade	Large-scale graphene manufactured for industrial use is referred to as industrial grade graphene. Usually, it is premium graphene that satisfies requirements for purity, layer thickness, and other characteristics. Because of its remarkable qualities, industrial-grade graphene is employed in many industries, including the automotive, electronics, and energy sectors.
Research Grade	Graphene created and utilized particularly for study is referred to as graphene research grade. It is frequently used in research studies to investigate the special qualities of graphene and its possible uses in a variety of industries, such as electronics, materials science, and biomedicine. Generally, it is of high quality and has few faults.
Technical Grade	Technical grade graphene is defined as graphene that satisfies particular quality and purity requirements and is appropriate for use in technical and industrial settings. It is commonly distinguished by its exceptional purity,

reliable quality, and particular qualities made for usage in a range of industrial processes and goods, such as electronics, composites, and coatings.

3.2.3 Zirconia

In an effort to improve lubrication and lower friction in vehicle engines, zirconia nanoparticles (ZrO_2) were being investigated as additions for treated engine oil. Zirconia nanoparticles can increase the longevity and efficiency of engine components, resulting in smoother operation and possibly a longer engine life because of their high hardness and temperature stability. Because these nanoparticles lower frictional losses, they can also improve fuel efficiency. Zirconia nanoparticles may also help with greater heat dissipation and maybe lower engine temperatures by enhancing the oil's thermal conductivity. When added to treated engine oil, zirconia additives have the potential to improve engine longevity and performance overall. The properties of zirconia are shown in Table 3.5.

Table 3.5 Properties of Zirconia (EU et al., 2021)

Properties	Value
Color	Grey
Composition	Yttria Stabilized
Density	5.7 g/cm^3
Young Modulus	200 GPa
Fracture Toughness K_{IC}	17 MPa $m^{1/2}$
Hardness	13 GPa
Maximum Temperature	1500
Flexural Strength	1000 MPa
Compressive Strength	2000 MPa

3.3 Filtration of Waste Engine Oil

The important process involved to treat the waste engine oil was filtration. Using ASTM D-7317, the waste engine oil was filtered to get the treated engine oil then move to next process which is combination with the additives. The apparatus of filtration is shown in Figure 3.2. Vacuum pump also needed to make the filtration process run quickly.

Figure 3.2 Filtration Apparatus (ASTM D-7317)

3.3.1 Filtration Process ASTM D-7317

A technique for assessing filter element performance by gauging how well they remove impurities from a waste engine oil is described in ASTM D7317. This standard is essential for ensuring that filters fulfill certain cleanliness standards in sectors like aerospace and automotive where filtration is vital. This method involved filter the waste oil using filter paper to get the coagulated pentane insoluble in the waste engine oil.

The process was started by preparation of the glass funnel, anodized aluminum clamp, sintered glass support base, ground joint flask and vacuum pump as part of the filtration system.

The apparatus was in dry and clean. To fit the filter holder, the filter paper was cut to the suitable size. Filter paper was placed between glass funnel and sintered glass support base. For better result, 2 filter paper were used at one time. After all apparatus were fitted nicely, the vacuum pump was joint to the sintered glass support using a tube to make the filtration process faster. The filtration process was done three times. By the end of this experiment, around 500mL the treated engine oil was be collected to combine with additives by next process.

3.4 Graphene and ZrO_2 Nanoparticles Preparation

Enhanced thermal and electrical conductivity was provided by graphene and zirconia additives in treated engine oil, which lower wear and increase fuel efficiency. Additionally, they enhance the viscosity index, oxidation resistance, and oil stability, increasing the oil's useful life and guaranteeing steady lubrication over a broad temperature range, which enhances engine longevity and performance.

Several types of mixtures were used in this experiment. For sample 1, 100% of pure and unused engine oil SAE 5W-30 was used and being compared with others sample. Sample 2 until 5 was waste engine oil 100% that was filtered to discover the main reference for observation of the result of coefficient of friction (COF) and wear surface diameter (WSD) happen if some additives added to them. As for sample 2, the treated engine oil that being filtered was added with graphene industrial grade up to 0.25% to observe the change in result of COF and WSD. Graphene research grade was added in treated engine oil up to 0.25% for sample 3 followed by 0.25% of graphene technical grade for sample 4. This experiment was used 3 grades of graphene to find if there any difference in COF and WSD. For sample 5, the treated engine oil was added with zirconia to find out the COF and WSD result. Lastly sample 6 was used engine oil SAE 5W-30 without any process or additives involved. Table 3.6 shows the sample percentage (volume/volume) of each sample.

Table 3.6 Sample Percentage (volume/volume) of Nanoparticles and Engine Treated Oil

No of sample	Oil	Percentage Oil (%)	Additives (%)
1	New engine oil SAE 5W 30	50	0
2	Filtered engine oil	49.875	Graphene industrial: 0.125%
3	Filtered engine oil	49.875	Graphene research: 0.125%
4	Filtered engine oil	49.875	Graphene technical: 0.125%
5	Filtered engine oil	49.875	Zirconia: 0.125%
6	Waste Engine Oil SAE 5W-30	50	0

The calculation for the additives in each sample were decided by formula mass (mg) = density (g/ml) x volume (ml). The mass of the additives in each sample were calculated by density of each additive's times with volume of the additives which were 0.125%. Each additive has different density. Table 3.7 shown density for each additive.

Table 3.7 Density of Additives

Additives	Density (g/ml)
Graphene Industrial	0.6
Graphene Research	0.06
Graphene Technical	0.45
Zirconia	6.05

The formula above was used after the density of each additive was known to find the mass of the additives that be need to mix with the filtered engine oil. The calculation was as shown in Table 3.8 below.

Table 3.8 Calculation of Mass of Additive

Additive	Calculation (density x volume)	Mass (mg)
Graphene Industrial	0.6 g/ml x 0.125 ml	75
Graphene Research	0.06 g/ml x 0.125 ml	7.5
Graphene Technical	0.45 g/ml x 0.125 ml	56.25
Zirconia	6.05 g/ml x 0.125 ml	756.25

3.5 Mixing Homogenizing Ultrasonic Process

In order to make a homogeneous mixture, the graphene and zirconia were mixed with the filtered engine oil using the homogenizing ultrasonic process. Ultrasonic waves were high-frequency sound waves that are above the range of human hearing, usually above 20 kHz will was used in this experiment. The temperature of the sample was kept under control during the homogenizing process, not going above 70°C. After that, 20 minutes at 50% amplitude and 0.5 active interval were spent using an ultrasonic homogenizer (Sartorius Labsonic P). 0.3 vol.% of a to prevent the nanoparticles from settling. Oleic acid, a surfactant, was added to the samples to help stabilize them.

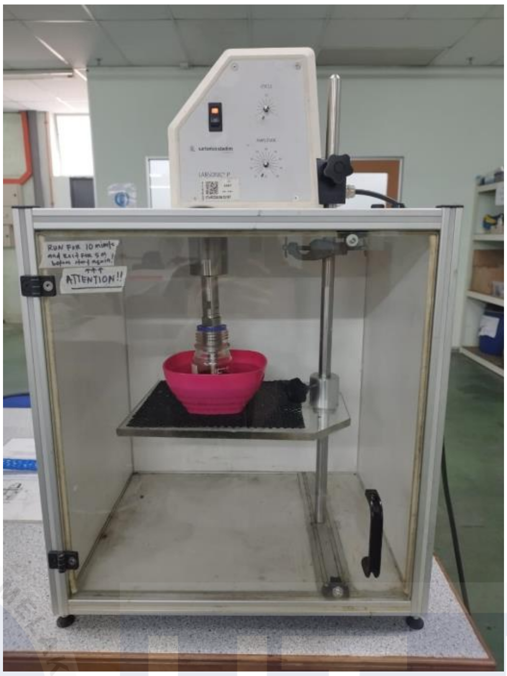

Figure 3.3 Homogenizing Ultrasonic Apparatus

Figure 3.3 above showed homogenizing ultrasonic apparatus. The process was held at Tribology Laboratory. It was started with preparation of the filtered engine oil and additives which were graphene and zirconia. It was important to choose a container that suitable with the chemicals that used. The nanoparticles are dispersed in a carrier fluid, which is often a compatible oil. This stage guarantees that the nanoparticles are evenly dispersed and suitable for mixing with the engine oil that has been filtered. Using an ultrasonic processor or homogenizer, the dispersion is exposed to ultrasonic waves. The fluid's cavitation bubbles, produced by the ultrasonic waves, cause significant localized heating and pressure changes. This contributes to the formation of a homogenous mixture by dissolving bigger nanoparticle agglomerates into smaller, more uniform particles.

Next, the engine oil that has been filtered was then mixed with the stabilized graphene and zirconia nanoparticle dispersion at the appropriate concentration, depend on the product and application. To guarantee that the nanoparticles are distributed uniformly throughout the treated engine oil, the mixture was vigorously shaken or agitated for a few minutes. Lastly, the sample where been tested by ASTM D-4172 apparatus. Graphene and zirconia nanoparticles should be added to filtered engine oil cautiously and in compliance with the manufacturer's instructions guidelines to prevent any detrimental effects on engine health or performance. This process was done 4 times, as 4 samples that mixed with additives needed for this experiment. Started with mix of filtered engine oil with graphene industrial grade, following by mix filtered engine oil with graphene research grade, next mix filtered treated engine oil with graphene technical grade. Lastly, mix filtered treated engine oil with zirconia.

3.6 Tribology Analysis ASTM D-4172 (4-Ball Tester)

The "Four-Ball Wear Test," or ASTM D-4172, was a standard test procedure used to ascertain the solid film lubricants, greases, and lubricating fluids' ability to resist wear under specified test conditions while they are in sliding contact. This test technique was frequently used to assess lubricants' performance in terms of their capacity to lower wear and friction in a range of mechanical systems.

The test involved the fourth ball is rotated against three steel balls in a lubrication cup with a predetermined force, speed, and duration are applied to the test. Following the test, the average scar diameter, which represents the lubricant's wear protection capabilities was determined by measuring the wear scar diameters on the three stationary balls. To ensure that the machine does not alter the data while operating, test conditions were an essential step in the process. Table 3.9 explain test condition that will be employ to create the precision data:

Table 3.9 Test Condition Range for ASTM D-4172 Process

Parameter	Range
Temperature	75 ± 2°C [167 ± 4°C]
Speed	1200 ± 60 rpm
Duration	60 ± 1 min
Load	392 ± 2N [40 ± 0.2 kgf]

3.6.1 Process of ASTM D-4172 (4Ball Tester)

The process was started by preparation with clean the four steel balls tester. These four steel balls were handling with care to avoid any damage to the balls that can affect the test result. For the lubricant's preparation, the 4 samples of filtered engine oil with additives that that have been processed by homogenizing ultrasonic mixing earlier were used and waste engine oil without any treatment process and also new engine oil SAE 5W-30. The sample must be around 2-4°C. The lubricant cup was filled with the lubricant and start with sample 1 which are new engine oil SAE 5W-30. The load as much 294.3 N. Speed for this test is 1200 rpm while duration is 60 minutes. Every lubricant was tested 3 times and the average of the result were taken.

The testing was started, three steel balls out of four were placed in the lubricant cup with the sample needed. The fourth test ball was mounted in the test machine's chuck or holder. 294.3N load was applied to the fourth ball. The fourth ball was rotated against the three stationary balls at a 1200 in 15 minutes. The eye must be kept on the test conditions to make sure they don't change during the test. The test balls were taken out of the lubrication cup after the test. The three stationary balls' wear scar diameters were examined with SEM. All 3 balls of scar reading will be collected to get the result Wear Scar Diameter (WSD). For Coefficient of Friction (COF), the 4-ball tester was generated the result. 1 sample were tested 3 times to get the average of COF. This test was done 18 times with different types of lubricants by referred to the 6 samples and 3 times for 1 sample.

3.7 Surface Roughness Using SEM

At high magnifications, Scanning Electron Microscopy (SEM) is a useful tool for examining surface shape and characteristics. SEM can offer useful information on surface topography and texture that can be coupled to roughness measurements obtained by other methods, including profilometry, even if it is not commonly utilized for direct surface roughness measurement.

SEM pictures which display characteristics like peaks, valleys, scratches and other surface imperfections can provide information regarding a sample's surface roughness. The surface's roughness qualitatively can be evaluating by examining these characteristics. Furthermore, surface characteristics like grain size, shape, and distribution may be measured using SEM pictures, and this information can be utilized to determine how rough a material's surface is.

For this research experiment, this SEM was used to gain the picture of wear scars on the steel ball from ASTM D-4712 process. Then, the result was gained and determined which lubricants give less wear to the balls bring mean that is the most suitable lubricant can be use as secondary lubricant. Figure 3.4 below shows the SEM apparatus.

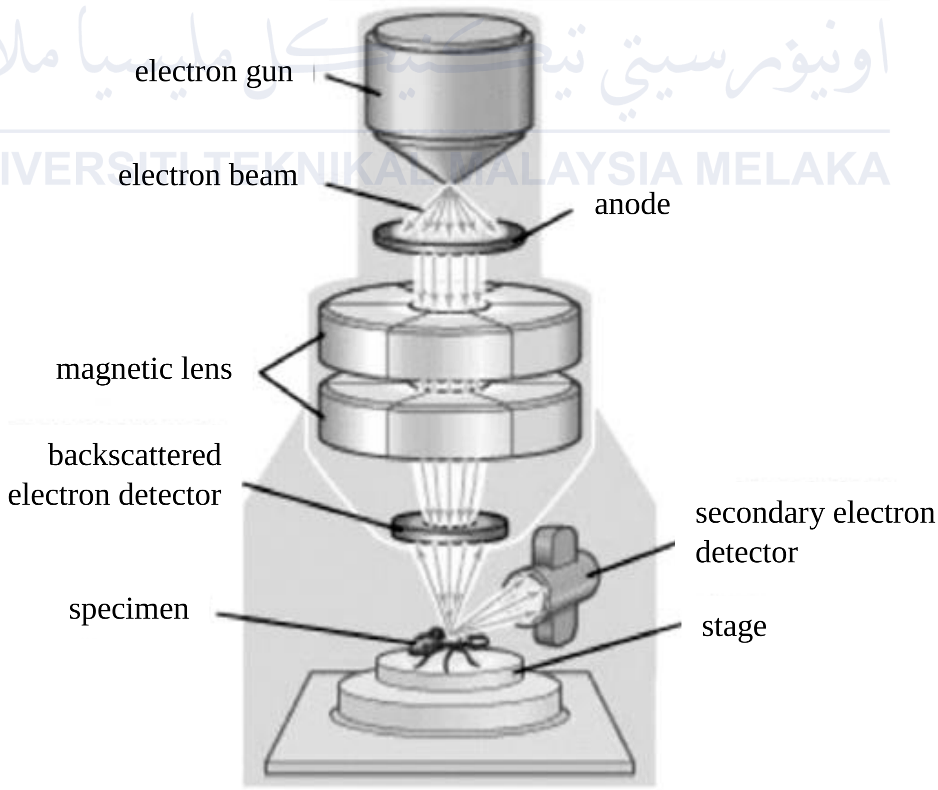

Figure 3.4 SEM Apparatus (Brittanica, 2023)

CHAPTER 4

RESULT

4.1 Introduction

In this chapter, the experimental data was scrutinized and analysed using the developed analytical method in Chapter 3. The causes were explored to identify the contributing factors to the acquired results for a thorough understanding of the outcomes based on empirical evidence. This work describes a new blend comprising filtered engine oil and two additives with a four-ball testing methodology based on ASTM D-4172 criteria in the pursuit of an improvement in tribological properties. The adopted analytical framework here assures methodical and consistent testing for which an analysis of performance is carried out in this experimentally designed setup of a tested combination and additives. The chapter is going to use this approach to bring out the possible benefits of the created formulation, its tribological properties, and applicability for a range of uses in view of the ASTM D-4172 requirements.

This is supposed to bring out the potential of additives in enhancing lubrication properties of reused engine oil at cost-effective and sustainable solutions for industries. Therefore, SAE 5W-30 gave the least coefficient of friction COF, among the six samples tested and is taken as a benchmark. Among the additives, graphene industrial-grade filtered waste oil performed best, having a COF closest to that of new engine oil, due to its better homogeneity of the particles and film-forming characteristics. Zirconia proved to be a good friction modifier; the reduction in COF was lower compared to graphene industrial grade. Graphene research-grade and graphene technical-grade reduced friction but were less effective and inconsistent. The highest COF was found with the untreated waste oil, indicating how urgent the additive treatment was before reusing it. These results, therefore, emphasize the optimization of the additive selection to develop better lubrication, wear reduction, and industrial sustainability.

This research exposed the performance of various additives improving anti-wear property-treated waste engine oil and that the graphene industrial grade turned in the best performance for attaining the lowest WSD of 0.2023mm, followed closely by zirconia at 0.2033mm. Graphene of research grade was also highly enhanced; though technical-grade graphene was less effective, it still outperformed untreated waste oil. Waste oil without additives had the poorest wear resistance, with an WSD of 0.2420mm, indicating that treatment is absolutely necessary. Additives such as graphene and zirconia enhance lubrication by forming a protective film on the metal surface, hence reducing friction, wear, and energy losses. These results confirm the effectiveness of advanced additives for the reinvigoration of waste oil for sustainable industrial applications, with minimal environmental impact and in tune with energy-saving practices, besides depicting the superior anti-wear property of commercial engine oil SAE 5W-30.

The SEM analysis and WSD measurements have been done to present a detailed assessment of lubrication performance and wear mechanisms of different engine oils. SEM images show the surface morphology and wear patterns on steel balls tested with different oils, showing the wear mechanisms such as an abrasive and adhesive wear. New engine oil (SAE 5W-30) has the least surface damage with grooves of similar depth, which suggests moderate wear and good lubrication. In contrast, much deeper grooves, pits, and generally rougher textures are seen for the untreated waste engine oil, which reflects its deteriorated lubrication property and higher wear. Smoother surfaces and reduced wear are observed for treated waste oils with additives, like graphene research grade, which reflects an improved tribological performance. Grooves, micro-pits, and material transfer point to active protection by additives in wear reduction. Still, these residual wear marks testify to the fact that lubrication performance was improved but not optimized. The combined SEM and WSD data illustrate the potential role of additives such as graphene to revitalize the waste oils to a sustainable industrial application.

4.2 Coefficient of Friction (COF) Differentiate

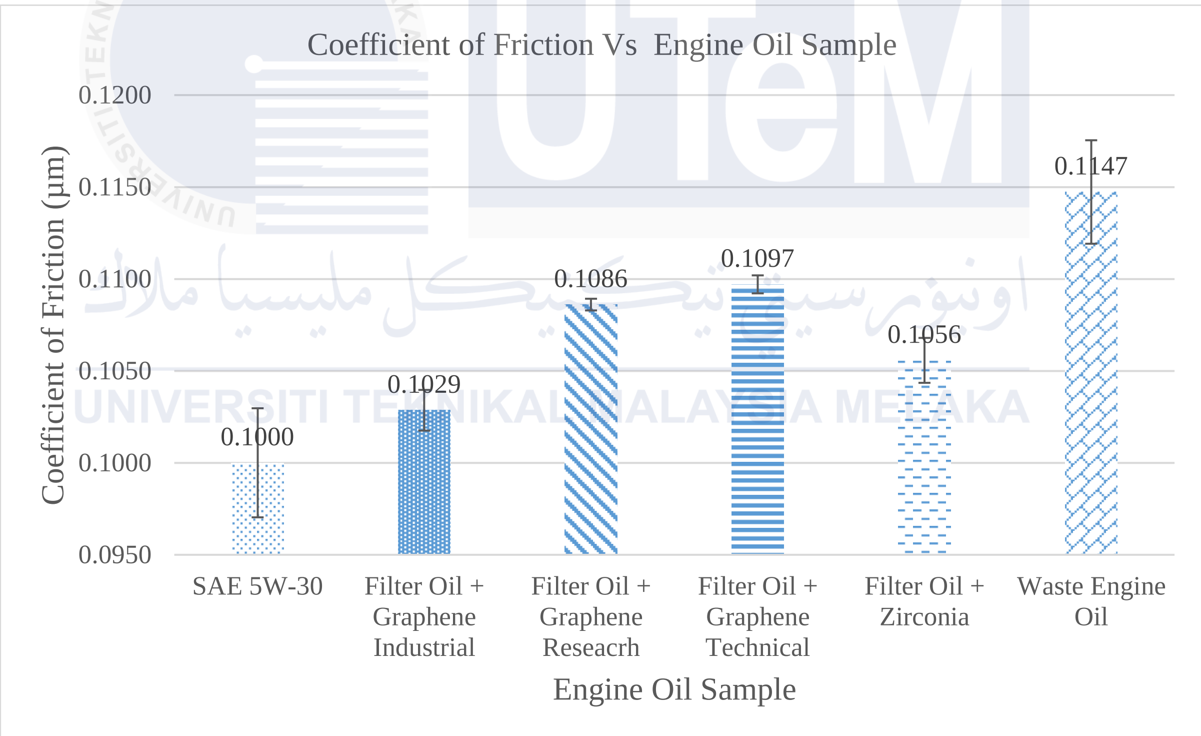

Figure 4.1 Coefficient of Friction Vs Engine Oil Sample Graph

Figure 4.1 above shows the COF result of testing involving 6 samples with different oil type. The results showed a significant difference in the COF from the tested samples. The base SAE 5W-30 had the lowest COF averaging 0.100μm, reflecting its highest lubrication properties and was considered the base against which the treated oil samples were compared.

When filtered oil mixed with graphene industrial grade was tested in 4-ball tester process, the COF was 0.1029µm, hence establishing the fact that an additive in the oil can reducing friction while maintaining properties relatively similar to that of the base oil. Next came the mixture between filtered oil and graphene research-grade, with a higher COF of 0.1086µm, showing less effectiveness in the reduction of friction. Graphene technical grade yielded a COF of 0.1097µm and proved to be the poor-performing oil among the tested graphene additives. Nevertheless, all graphene treated samples performed better compared with untreated waste engine oil.

The COF for the filtered oil with zirconia is superior to that resulting from the filtered oil in graphene research and graphene technical grades, since zirconia can form a stable and wear-resistant layer on contacting surfaces. Zirconia was nano-sized ceramic particles as lubricant and the overwhelming preponderance of additives has positive tribological attributes with non-metallic superficial materials (Kuti et al., 2022). However, it lacks the great lubricity provided by graphene industrial grade, which is specially engineered for superior tribological performance. Graphene industrial grade most probably has a more homogeneous particle size, higher purity, and better dispersion in oil than graphene research and technical grades; therefore, it could build a smoother and more effective lubricating film. This superior film formation minimizes surface asperities and thus reduces friction more effectively compared to zirconia, which explains why the COF of filtered oil with graphene industrial grade is lower than that of filtered oil with zirconia.

Among all, the zirconia additive proved to be a promising alternative friction-reducing agent, attaining a COF of 0.1056µm. It didn't perform like graphene industrial grade, yet it outperformed both graphene research and technical grades, hence proving that this additive could be cost-effective and efficient in improving lubrication. On the other extreme, the untreated waste engine oil had the maximum COF averaging 0.1147µm, thus reflecting its poor performance on tribological aspects, while requiring additive treatment before reutilization in industries.

These results depict the possibility of treated engine oil combined with additives to enhance its tribological characteristics for industrial applications in a sustainable manner. Additions of graphene industrial grade gave the best solution and performance closest to SAE 5W-30. Zirconia presented a good potential to reduce friction, thus presenting an alternative to graphene additives. The high COF of the untreated waste engine oil strengthened the need for reconditioning and additive treatment for reuse in industries. In general, this study emphasizes the importance of optimization in additive selection to improve the performance of treated oils, enabling sustainable practices in lubrication and wear reduction for industrial machinery.

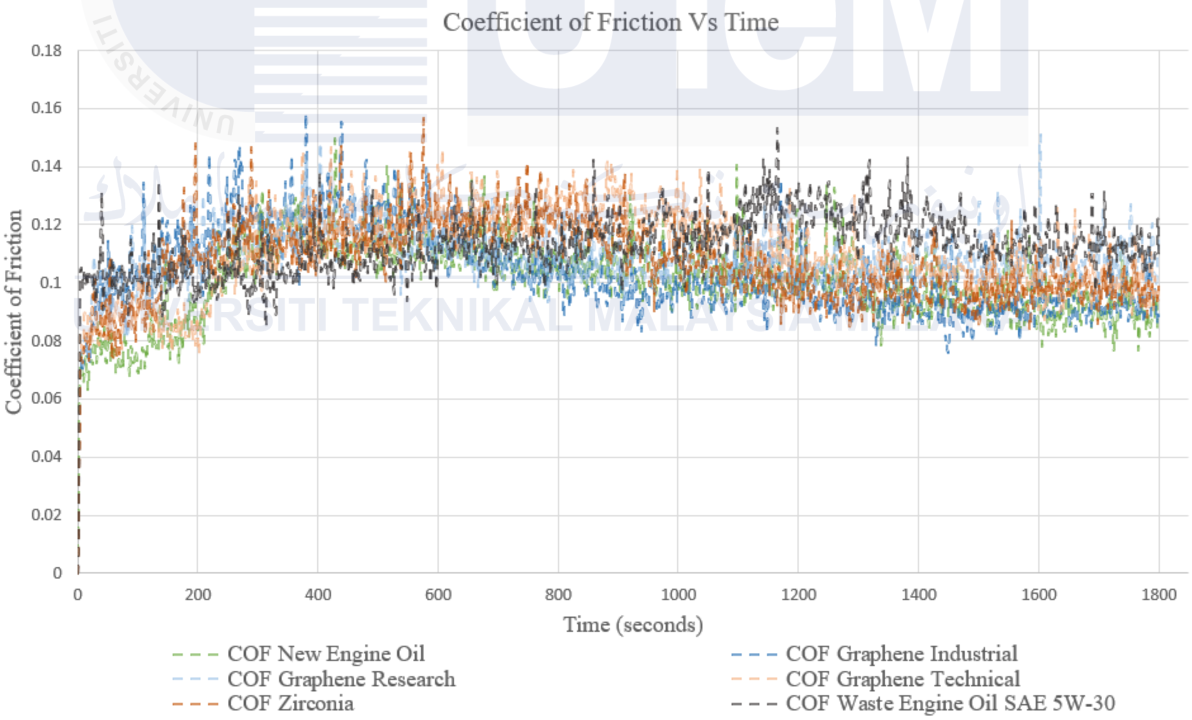

Figure 4.2 Coefficient of Friction Vs Time Graph

Figure 4.2 below is graph generated from 4-Ball Tester result to shows that all the filter engine oil with additives can be as good as new engine oil in COF. Lower COF means less friction between engine components. Reduced friction minimizes energy losses, hence better engine efficiency and fuel economy. With less friction, there is less mechanical wear on the engine parts like pistons, cylinders, and bearings, which prolongs the life of the engine components and reduces maintenance costs. Low COF oils help to provide for smooth and continued operation of engines, preventing overheating problems and ensuring optimal performance under high stress or load conditions. Friction also produces heat. By lowering COF, engine oils make the engine run cooler; the thermal stability of the oil improves, preventing potential problems like degradation of oil or fatigue in metals.

The COF vs. Time graph presents the frictional performance of various oil samples that have gone through the experiment in the 4-ball tester, underlining differences in lubrication effectiveness: new, waste, and treated oils with additives. During the running-in period, which is between 0-200 seconds, the new engine oil showed the lowest friction coefficient, proving its good lubrication characteristics. It stabilized in a very short time and remained constant during the test. All the other samples were compared with this one. Waste engine oil (SAE 5W-30) was the one that showed the highest COF and large oscillations; it was tested without additives and presented the worst lubrication quality among the tested ones because of its degradation.

Filtered waste engine oil with the addition of additives showed remarkable improvements in frictional behaviour. Among the graphene-enhanced samples, the industrial-grade graphene additive showed the lowest COF and its variation trend was stable, close to that of new engine oil, which could suggest its effectiveness in friction reduction. The research-grade graphene also had an improved lubrication performance but with a slightly higher COF than the industrial grade, indicating it was marginally less effective. The technical-grade graphene had higher COF values and larger standard deviations, which indicated that it was less capable of maintaining consistent lubrication. Another additive, zirconia, demonstrated a significant reduction in COF compared to untreated waste oil, with slight fluctuations during stabilization, hence showing promise as a friction modifier.

In general, the study has shown the potential of additives in enhancing the lubrication properties of reused engine oil for industrial applications. Of the tried adducts, industrial-grade graphene proved to be the best additive, giving performance closest to new engine oil. Anti-wear, anti-oxidant, dispersant, detergent, friction modifier, viscosity modifier, extreme pressure agent, antifoaming agent and emulsifier compounds are the most common additives (Nagy et al., 2019).Among other additives, research-grade graphene and zirconia show positive effects, though remaining a little inconsistent in performance. These results highlight the possibility of waste engine oil reuse, with the help of advanced additives for treatment, providing cost-effective and sustainable solutions to satisfy industrial lubrication needs.

4.3 Wear Scar Diameter Differentiate

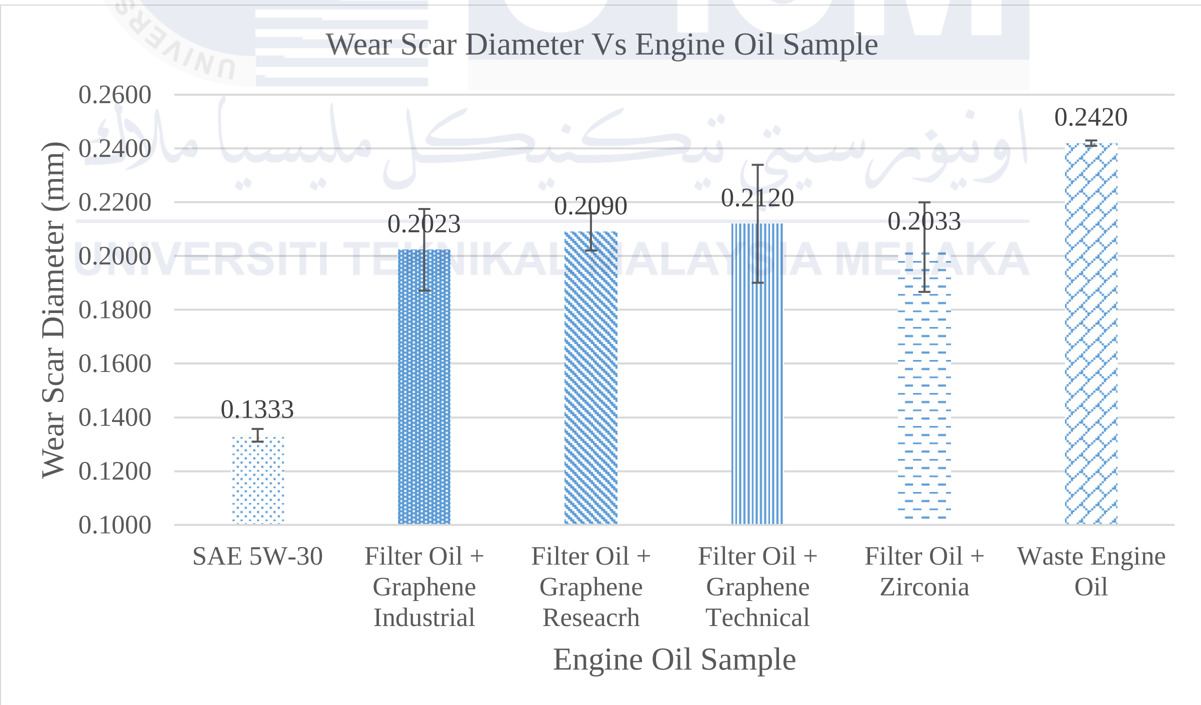

Figure 4.3 Wear Scar Diameter Vs Engine Oil Sample Graph

Figure 4.3 shown the result of Wear Scar Diameter of 4-Ball Tester in 6 samples with different types of oil. Sample 1 has the lowest WSD of 0.1333mm and hence far better than all other samples. It was because anti-wear additives like zinc dialkyldithiophosphate and friction modifiers added to this grease create a boundary layer on the metal surfaces to resist wear. Thus, it can provide very good wear resistance, SAE 5W-30 maintains its engine for a long duration with better overall performance when used in operating conditions. The results confirm that commercial engine oils such as SAE 5W-30 were formulated with the express purpose of minimizing friction and wear to reduce mechanical damage and energy losses.

The result in Sample 2, filtered engine oil with graphene industrial grade additive really highlights the enormous leap in improvement within its anti-wear performance. This was well evidenced by a reduced Wear Scar Diameter (WSD) of 0.2023mm. Such an improvement underlines graphene's role in reducing friction and wear through building up a protective film on the contact metal surfaces under load. This can be attributed to the higher purity and consistent structural properties of the industrial grade graphene ensuring the most appropriate dispersion in the oil matrix and uniform interaction with the metallic contact areas. These attributes were vital for re-used industrial oil applications since maintaining or improving lubrication performance is one of the key factors to prolonging equipment life and reducing operational costs.

Next results were obtained by adding zirconia to the filtered engine oil with an WSD of 0.2033mm in comparison with graphene industrial grade. Zirconia represents a ceramic material with good thermal stability and wear resistance. It enhances the re-used engine oil's properties in tribology thus offering more lubrication rather than metal-to-metal contact. This reduction in wear underlines its potential as a cost-effective and efficient alternative to graphene in recycled oil formulations. Given the properties of zirconia, this work develops an understanding of how commercialized and naturally derived additives can further optimize friction and wear characteristics of treated engine oils for sustainability in industrial applications. This insight goes into the bigger perspective of decreasing dependency on virgin lubricants through the extension of functional life for used oils.

Next the WSD obtained which were 0.2090mm. This infers that the addition of the graphene additive of research grade could significantly enhance the wear protection characteristics of the engine oil. The improved performance was believed to result from the homogeneous structure and further good dispersion within the oil matrix, enabling possibly the better tribology formation on metal contacts under load. Compared to technical grade graphene, which presented a higher WSD, research grade graphene had refined structural properties and was more compatible with the oil showing its effectiveness in minimizing friction and wear. This finding realizes the potential of research grade graphene to serve as an economically viable and sustainable additive that can improve performance in reused industrial oils. This underlines the purpose of the study: comparing the efficiencies of commercialized and organic additives in friction reduction and wear prevention.

The higher WSD of 0.2120mm for the technical grade graphene additive shown that material purity and consistency are very important to achieving optimal lubricity. While the technical grade resulted in lower performance compared with industrial and research grade graphene additives, it still showed significant improvement compared to untreated waste engine oil and therefore proved to be a very effective additive in the re-purposing of industrial oils. This could be considered an indication that impurities or inconsistency in the technical grade material interfere with the material's ability to form a stable, wear-protective film under load. A finding of this nature is important in assessing the economic and practical feasibility of different grades of additives since such findings can reveal the trade-offs among cost, material quality and performance. This study has pointed out the additive refinement of waste oil to its best re-usage, which is quite aligned with sustainable industrial practices.

The maximum WSD value of 0.2420mm for waste engine oil SAE 5W-30 without any additive showed its poor anti-wear characteristic compared to other samples. This result indicated that during long use, contamination, and oxidation processes, this oil lost much of its lubricating characteristics. With time, the accumulation of debris and chemical interactions with oxygen along with thermal stresses exhaust the capability of the oil to decrease friction and wear. High value of WSD indicated not only poor performance of waste engine oil but a serious demand for its effective treatment and additive enhancement too. This might allow waste oil to be revitalized with advanced additives or filtration processes, restoring anti-wear properties and offering a sustainable approach to industrial reuse that could reduce environmental impact.

These results confirm that additives such as graphene and zirconia are very important to improve the anti-wear property of treated waste engine oil. Graphene is also found to be one of the most promising lubricating additives in tribo-logical applications. It has been proved that lubricating contacting surfaces with lubricant containing graphene materials as additives exhibited superior anti-frictional and anti-wear performances, which result in energy savings, low carbon emissions, and less wear and tear of mechanical components (Dhanola & Gajrani, 2023). Among the graphene additives, industrial grade graphene showed the best performance. This could be because of the optimization in particle structure and its superior dispersion characteristics that would provide better performance in friction and wear reduction. Research grade graphene followed suit with a slightly lesser efficiency but still an impressive improvement over the plain waste oil. Technical grade graphene was less effective compared to its research and pristine grades but still showed notable enhancements further demonstrating the adaptability of graphene as an additive. Zirconia also had similar performance proving to be a promising alternative with high thermal stability and wear-resistant properties. Improvement of the lubricating characteristics of the reclaimed oil by these additives evidences their potential in providing sustainable lubrication solutions able to enable waste oil reutilization while maintaining the industrial standards.

The better performance of SAE 5W-30 underlines the efficiency of commercial formulations concerning friction reduction and wear. On the other hand, additive use in treated waste oil provides an environmentally friendly and economical solution for industrial applications by reducing waste and preserving resources. These findings support the feasibility of reusing treated waste oil with appropriate additives, contributing to sustainable practices in the automotive and manufacturing industries.

4.4 Surface Morphology Analysis

The SEM images and wear scar diameter measurements will be important in this project to analyze the performance of filtered engine oils. These SEM images provide a critical display of the surface morphology of the wear scars on the test balls which allows understanding of the wear mechanisms at the microscopic level. The wear scars of the balls subjected to new engine oil may have minimal surface damage as a result of its higher lubrication characteristics. While the untreated waste engine oil may show major scarring and surface irregularities indicating a higher wear.

In the case of treated engine oils, the SEM images will point to the effects that additives caused on the wear scar surface. As such filtered engine oil with graphene research grades may show smooth wear scar surfaces and material transfer reduced in comparison to an untreated waste engine oil.

Quantitatively, these observations are confirmed by the WSD measurements. The smaller the MWSD, the better the lubrication performance and the less the wear. In the present work, new engine oil should provide the smallest WSD followed by filtered oil with additives research-grade graphene. On the contrary, untreated waste engine oil is expected to exhibit the biggest MWSD due to deteriorated lubrication properties. The combination of SEM analysis with MWSD data will help elucidate the full picture with respect to the efficacy in wear reduction and reusability improvement of treated waste engine oil for industrial applications.

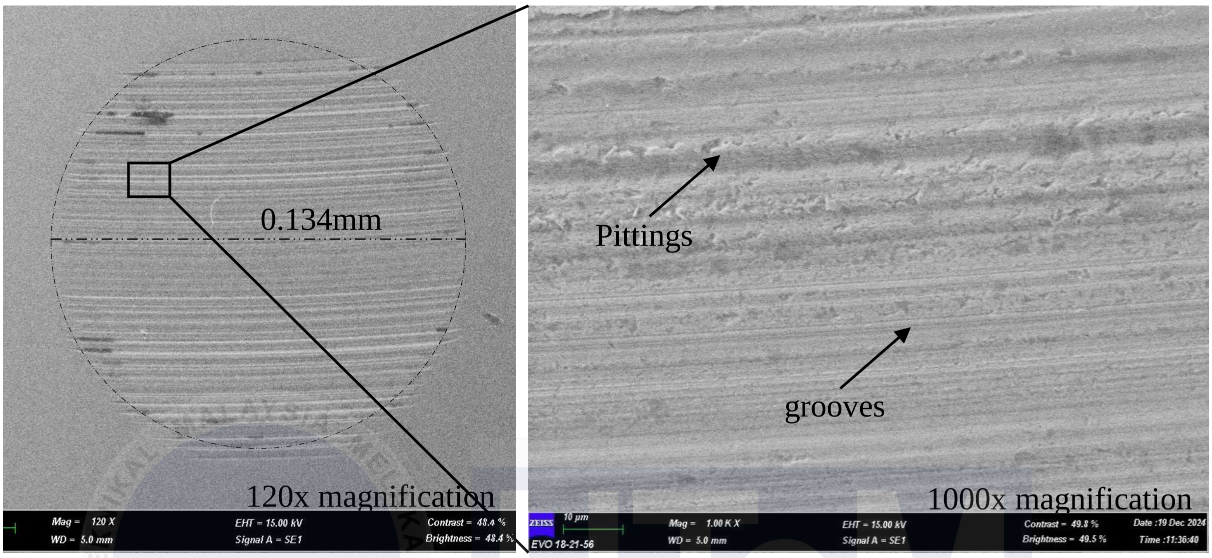

Figure 4.4 SEM Image for New Engine Oil SAE 5W-30

From Figure 4.4 above, the provided SEM images of the steel ball tested with new engine oil SAE 5W-30, magnified at 120x and 1000 magnification, wear patterns are evident on the surface. The wear track at low magnification appears as parallel grooves across the surface of the steel ball. Most likely, these grooves are caused by abrasive wear due to hard particles or asperities within the oil or in the material of the steel ball itself scraping along the surface. The pattern indicates consistent sliding motion during the test, which is quite normal in 4-ball tester operations.

In comparison, the grooves are deeper, and with high magnification, it's possible to observe other features such as micro-pits or small scratches. Such features are probably due to adhesive wear, characterized by localized welding and material transfer between contact points under high pressure. The surface roughness shows the existence of both mechanisms of abrasive and adhesive wear.

A continual groove and roughness imply there is a certain moderate wear but no catastrophic failure regarding crack occurrences or high rates of material removal. Indeed, this wear looked rather uniform to show that this new SAE 5W -30 engine oil should maintain some reasonable lubrication though inadequate film strength with possibly existing abrasive particles in such types of oils contributes to these observed wear marks.

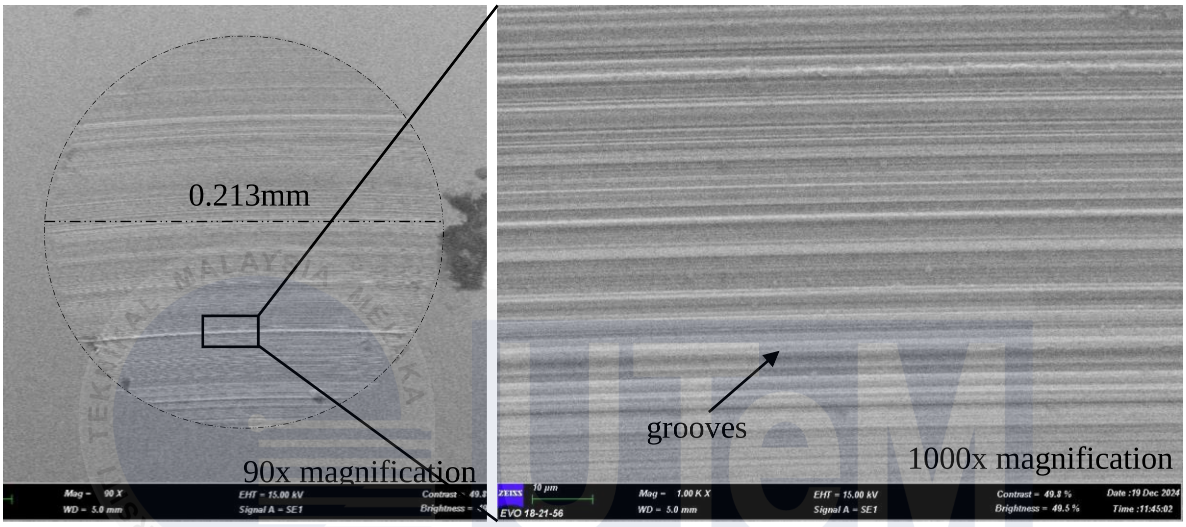

Figure 4.5 SEM Image for Filtered Engine Oil with Graphene Research

Based on the Figure 4.5, in the first image which is 90x magnification there is a prominent wear scar representative of the area in which sliding contact occurred during the 4-ball test. The scar includes grooves and surface irregularities-symptoms that are quite characteristic of wear resulting from friction and load. The nature of the appearance of such features testifies to material removal through adhesive and abrasive wear mechanisms.

The wear track is much more clearly revealed in the second image at the higher magnification of 1.00k times. There are parallel striations in the direction of sliding, pointing to material ploughing, probably caused by micro-abrasion or by hard particles moving around trapped between the sliding surfaces. Such features provide evidence of ongoing interaction between the surface and lubricating oil modified with graphene research grade additives.

The wear is evident but the relative uniformity of grooves and the lack of heavy surface deformation indicate that this additive did contribute to the reduction of extreme wear by protecting a layer though it not eliminate it entirely. The wear patterns therefore carry useful information on the performance of the lubricant with the additive under test conditions.

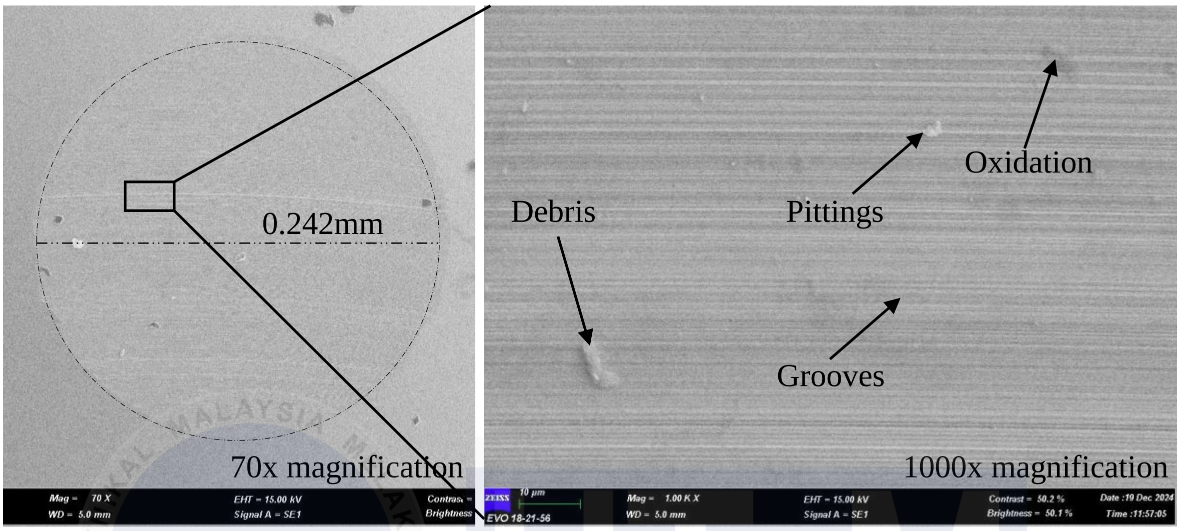

Figure 4.6 SEM Image for Waste Engine Oil SAE 5W-30

Figure 4.6 shows that SEM image 70x magnification showing overall wear scar morphology. In this low magnification, the wear scar appears quite circular. It suggests the point of contact between the ball bearings during the test. Some visible striations are observed across the surface of the wear scar. These striations indicate a sliding contact, a very common wear mechanism where one surface slides over another with some kind of insufficient lubrication. The various pits and dark areas in the figure may also be identified, which could well have been due to abrasive wear or localized damage because of the presence of debris particles in waste oil. Abrasive wear occurs when hard particles or contaminants in the oil become trapped between contact surfaces and cause material removal. The relatively uneven surface and darker regions testify to the compromised performance of the lubricant, probably due to degraded properties of the waste oil.

The image with higher magnification of 1000x magnification shows in detail the surface features within the wear scar. Some grooves and scratches observed here confirm the presence of abrasive wear. Features that indicate the particulate contamination level in the waste oil is high, and thus this acts as a cutting agent to accelerate the wear process. Moreover, any small deposits or patches seen on the surface could represent products of oxidation or residues resulting from the degraded oil. Commonly, waste oil consists of combustion by products, degraded additives and metal particles which degrade the quality of lubrication. The rough texture with visible micro-pits also suggests adhesive wear, where there is material transfer between the surfaces because of high local friction and poor lubrication. This generally causes material tearing and surface roughening. The picture also shows pitting, the small, localized depressions or holes that may be caused by wear, corrosion, or material fatigue.

CHAPTER 5

CONCLUSION AND RECOMMENDATION

5.1 Conclusion

This research was carried out to study and compare the wear behavior and lubrication performance of waste engine oil (SAE 5W-30) with new engine oil and waste oils filtered with different additives. The main objectives of the research, as outlined in Chapter 1, were to explore the coefficient of friction and mean wear scar diameter by using the 4-ball tester method, assess the efficiency of graphene and zirconia as additives, and find the best formulation which would provide the highest improvement in lubrication properties.

It can be seen from the results that waste engine oil showed severe wear and poor lubrication characteristics compared to other samples. SEM analysis of worn surfaces at 70x and 1.00kx shows pronounced grooves with severe surface damage. Such characteristics were observed to be remarkably poorer compared to those in virgin new engine oil and additive-added filtered oils. Waste engine oil with a higher measured COF and larger MWSD further attests its poorer performance.

In turn, industrial, technical, and research-grade filtered oils with graphene additive, as well as with zirconia, showed better lubrication characteristics. Among the samples tested, the industrial-grade graphene additive was the best in terms of wear and friction reduction and thus proved the efficiency of nanomaterials in improving the characteristics of engine oil. The objective of the paper was fully met since it allowed establishing a full comparison of the lubrication characteristics for different samples of oil and underlined the role of advanced additives in wear and friction reduction.

5.2 Recommendation

It is not advisable to use waste engine oil in applications needing high lubrication performance, as obtained from this research work due to its severe wear effects. The industries should, therefore, consider incorporating advanced additives like graphene or zirconia into recycled or filtered oils to improve their properties. Further studies shall be targeted at optimizing the concentration of these additives to maximize their effectiveness in wear and friction reduction. Further research into the long-term stability and performance of these additives at different temperatures and pressures would better simulate field applications.

Recycling and reconditioning waste engine oil with additives not only upgrade its performance but also contribute to environmental sustainability by reducing oil waste and dependence on new production. Surface characterization by such advanced techniques includes the use of AFM and XRD for an understanding of wear mechanisms and nanoasperity interactions in great detail. Other nanomaterials, like MoS2 and boron nitride, could be tried out for the goal of determining effective additives in applications of concern. In a nutshell, this study underlines the potential of advanced additives to improve lubrication performance and, at the same time, offer sustainable solutions for reusing waste engine oil and furthering both industrial and environmental goals.

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APPENDICES

APPENDIX A. Gantt Chart Bachelor Degree Project 1

No	Subject		Week													
			W1	W2	W3	W4	W5	W6	W7	W8	W9	W10	W11	W12	W13	W14
1	Meeting with supervisor	Plan														
		Actual														
2	Decide project and process	Plan														
		Actual														
3	Write Introduction	Plan														
		Actual														
4	Discuss topic for methodology	Plan														
		Actual														
5	Write methodology	Plan														
		Actual														
6	Discuss literature review	Plan														
		Actual														
7	Write literature review	Plan														
		Actual														
8	Preparation proposal report	Plan														
		Actual														
9	Preliminary result	Plan														
		Actual														
10	Proposal correction	Plan														
		Actual														
11	Submission proposal report	Plan														
		Actual														

12	Presentation PSM 1	Plan														
		Actual														

APPENDIX B. Gantt Chart Planning of Bachelor Degree Project 2

No	Subject		Week													
			W1	W2	W3	W4	W5	W6	W7	W8	W9	W10	W11	W12	W13	W14
1	Meeting with supervisor	Plan														
		Actual														
2	Planning experiment	Plan														
		Actual														
3	Waste engine oil collection	Plan														
		Actual														
4	Filtration of waste engine oil	Plan														
		Actual														
5	Nanoparticle preparation	Plan														
		Actual														
6	Mixing homogenizing process	Plan														
		Actual														
7	Tribology analysis using 4-ball tester	Plan														
		Actual														
8	Surface analysis using SEM	Plan														
		Actual														
9	Data and result collection	Plan														
		Actual														
10	Writing result	Plan														
		Actual														

11	Writing conclusion and recommendation	Plan														
		Actual														
12	Proposal correction	Plan														
		Actual														
13	Submission proposal report	Plan														
		Actual														
14	Presentation PSM 2	Plan														
		Actual														